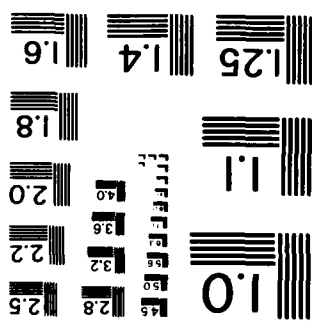


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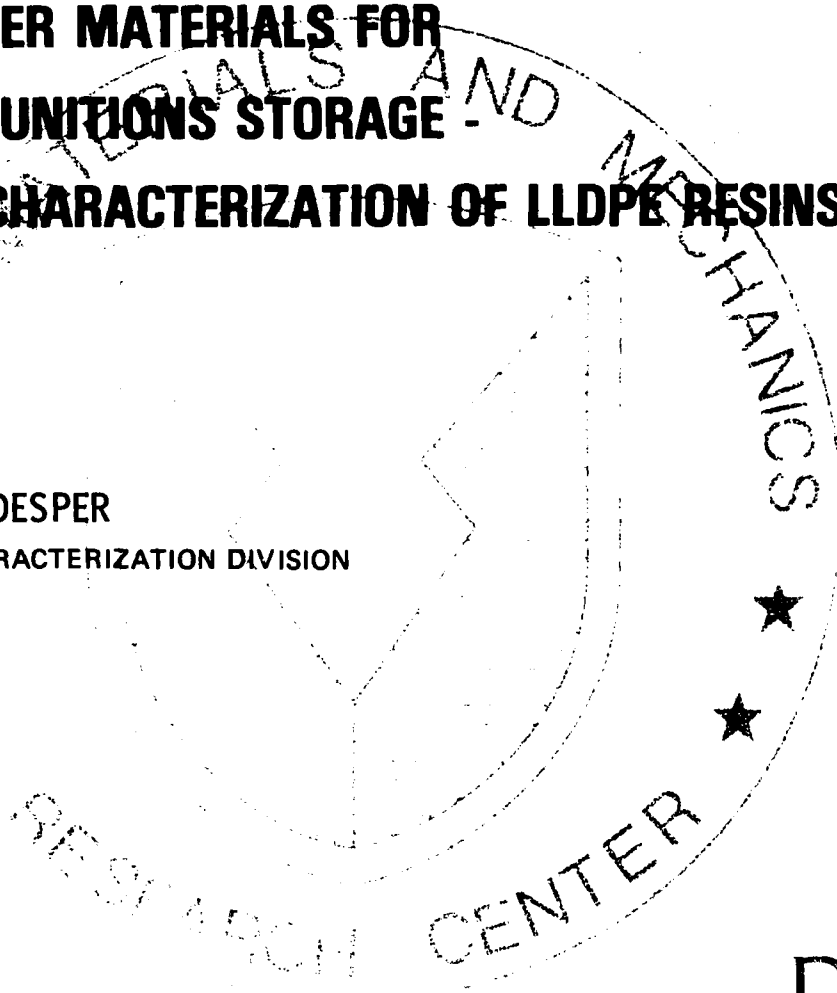




**CHARACTERIZATION AND SELECTION
OF POLYMER MATERIALS FOR
BINARY MUNITIONS STORAGE
PART 2: CHARACTERIZATION OF LLDPE RESINS**

C. RICHARD DESPER
MATERIALS CHARACTERIZATION DIVISION

July 1984



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ABSTRACT

This work is aimed at determining the materials properties required for storage of DF (methylphosphonic difluoride) over extended periods of time at temperatures as high as 70°C. Earlier studies showed the most likely mode of failure to be environmental stress cracking of the polyolefin container, rather than chemical or oxidative attack. The most promising candidates in terms of resistance to environmental stress cracking are ethylene copolymers with a small alpha-olefin comonomer content. The comonomer, typically butene-1 or hexene-1, introduces short side chain branches which disrupt the regularity of the crystal morphology, presumably leading to a greater proportion of intercrystalline ties. An X-ray diffraction method was developed for measuring the amount of short chain branching which offers greater accuracy than the previous infrared absorption method. Characterization of short chain branching by such a method, along with molecular weight characterization by gel permeation chromatography and crystallinity determination by density, were deemed essential to quality control of resins for the intended application.



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INTRODUCTION

The present work is a continuation of previously reported work¹ having the goal of determining the materials properties required for storage of DF (methylphosphonic difluoride) over extended periods of time at elevated temperatures. Attention has been focused on thermoplastic polyolefins, which, in some instances, appear to offer the requisite chemical resistance and long term stability. However, the performance of the current polyethylene container material and possible replacement materials must be critically assessed, particularly in view of new requirements for enduring more severe storage conditions.

The previous work¹ identified environmental stress cracking as the major factor contributing to premature failure in DF containers. This conclusion is in accord with the results of earlier screening studies by White and co-workers.² The literature on environmental stress cracking has been surveyed (see Appendix) and resin density, molecular weight, and applied stress are key factors influencing environmental stress cracking. As suggested in the previous work,¹ medium density is preferred to high density, since the greater crystallite perfection associated with high density in polyethylene would lead to a higher degree of regular chain folding instead of the more desirable intercrystalline links. Medium density is generally achieved by deliberate incorporation of a controlled amount of side chain branches in the polyethylene molecule by copolymerization with a small amount of an alpha-olefin (such as butene-1) which results in a short linear aliphatic side chain. High molecular weight is also desirable for better resistance to environmental stress cracking. The present work addresses itself to following up several recommendations from the previous work.¹

1. Development of a method for determination of side chain content from measurement of crystal lattice parameters,
2. Characterization of alternate of polyethylene resins as a possible replacement for the Marlex M407MQ used in the past, and
3. Development of a method for measurement of long chain branching for more complete resin characterization. The previous work also singled out residual stress, introduced by processing, as an aggravating factor leading to premature failure by environmental stress cracking. However, this factor shall be dealt with elsewhere as a processing problem and will not be addressed here, where the focus is on materials problems.

EXPERIMENTAL

X-Ray Diffraction Procedures

Data were obtained using a Xentronics Corporation XD10 area-imaging proportional counter mounted on a Picker four-circle goniometer.³ The primary X-ray beam is monochromated by reflection from a highly oriented pyrolytic graphite crystal and collimated

1. DESPER, C. R. *Characterization and Selection of Polymer Materials for Binary Munitions Storage*. Army Materials and Mechanics Research Center, AMMRC TR 83-45, August 1983.
2. WHITE, S. S., LEEP, D. A., FIELDER, D., and DUNNE, L. P. *The Permeation of the Chemical Components of Binary G-Munition Through Polyethylenes*. Edgewood Arsenal Technical Report EC-TR-74080, AD-C000378, Aberdeen Proving Ground, Maryland, November 1974.
3. DESPER, C. R., and BURNS, R. *An Area-Imaging Proportional Counter for X-Ray Diffraction*. *Adv. X-Ray Anal.*, v. 24, 1981, p. 161.

by a 0.5-mm circular pinhole aperture a distance of 225-mm from the X-ray source. The polymer film samples were placed in a special sample holder⁴ designed to hold a specimen in the reflection position as in a conventional Bragg-Brentano Powder Diffractometer. The area detector, placed at a distance of 290-mm from the specimen, covered a maximum angular range of 16° in both the horizontal and vertical directions.

The area detector rests on a detector arm which moves in a horizontal plane (see Figure 1), positioned to intercept the Bragg angle range of interest, chosen to be $2\theta = 14^\circ$ to 29° in the present instance. Data for this entire angular range were obtained simultaneously using the area detector in conjunction with a Wicat 150 mini-computer. Data for each pattern were stored in arrays of dimensionality 256 X 256. Two modes of data acquisition were used: (a) wide-range patterns ("Zoom I") were obtained covering the range $2\theta = 14^\circ$ to 29° with a vertical divergence range of $\pm 7^\circ$ (see Figure 2), and (b) more detailed patterns ("Zoom II") were obtained covering the range $2\theta = 20^\circ$ to 27° (see Figure 3) with a vertical divergence range of $\pm 3.5^\circ$. In both instances the instrument resolution was approximately 0.2° , but the data points were spaced at 0.054° and 0.027° intervals, respectively. The Zoom I patterns showed the entire range of interest, including the tails of the polyethylene amorphous peak, while the Zoom II patterns showed the shapes of the (110) and (200) crystalline lines in greater detail. The latter patterns were used for precise measurement of d spacings.

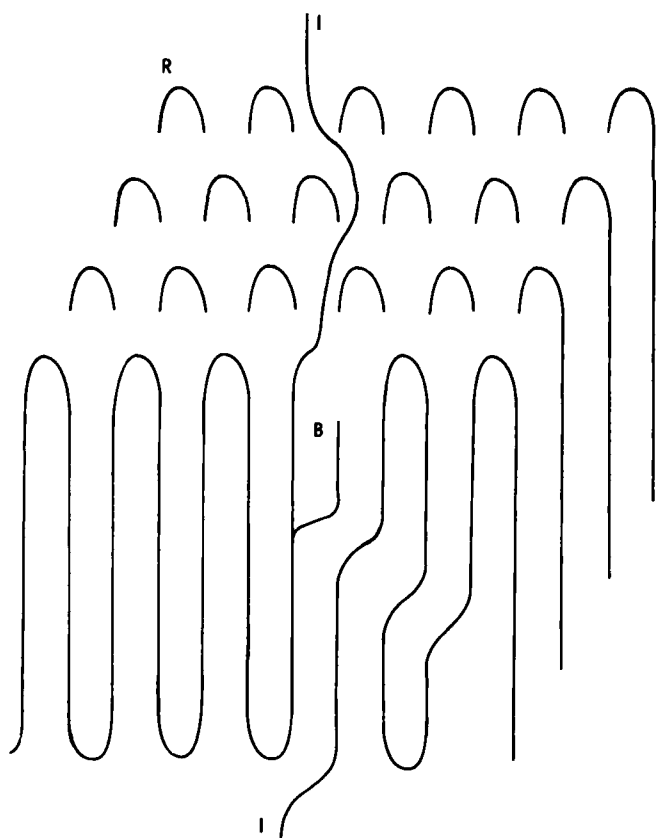


Figure 1. Polyethylene folded chain crystal showing intercrystalline links (I) resulting from a chain branch (B); also showing regular folds (R).

4. DESPER, C. R. *A Computer-Controlled X-Ray Diffractometer for Texture Studies of Polycrystalline Materials*. *Adv. X-Ray Anal.*, v. 12, 1969, p. 404.

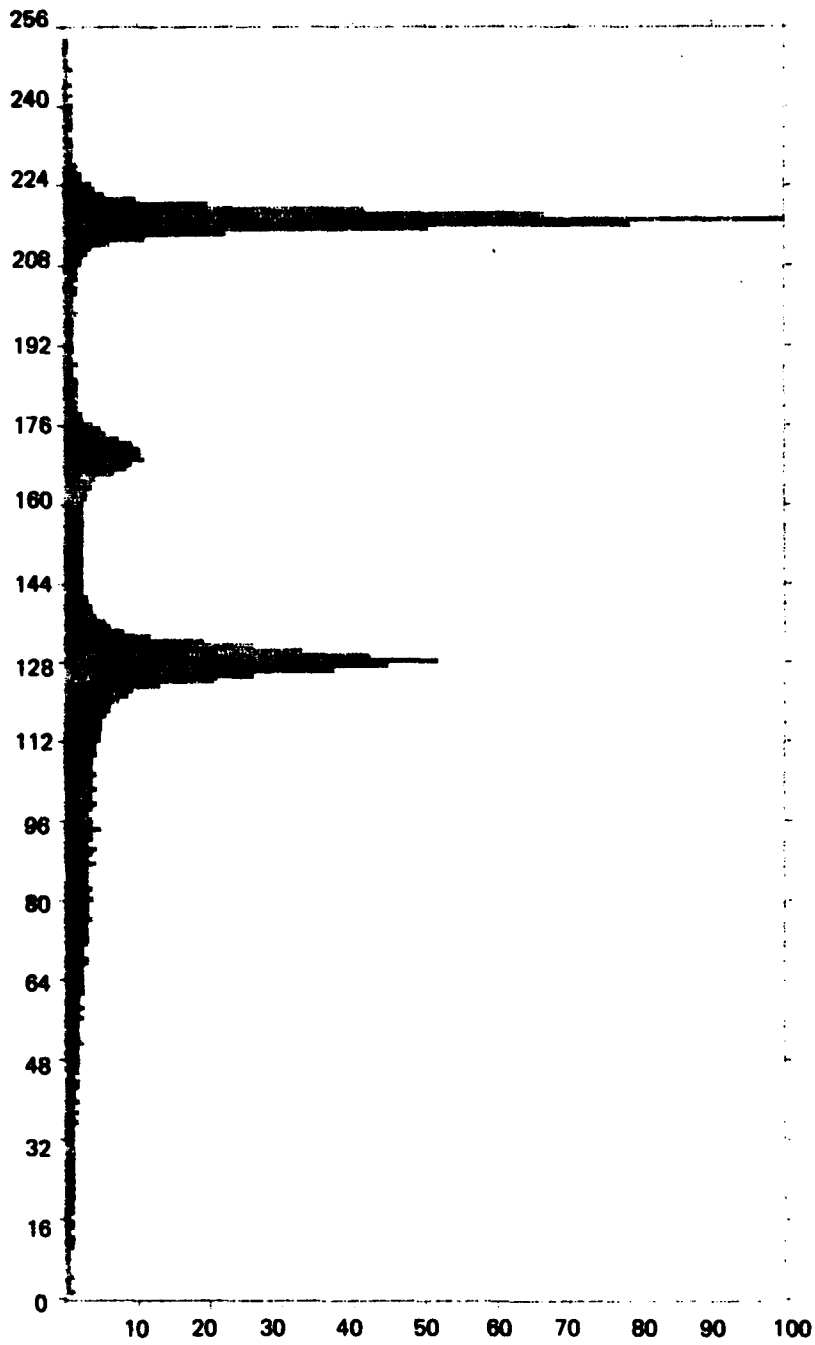


Figure 2. Diffraction pattern of M445 at Zoom I. Channel 128: polyethylene (110) reflection; channel 170: polyethylene (200) reflection; channel 218: graphite (002) reflection.

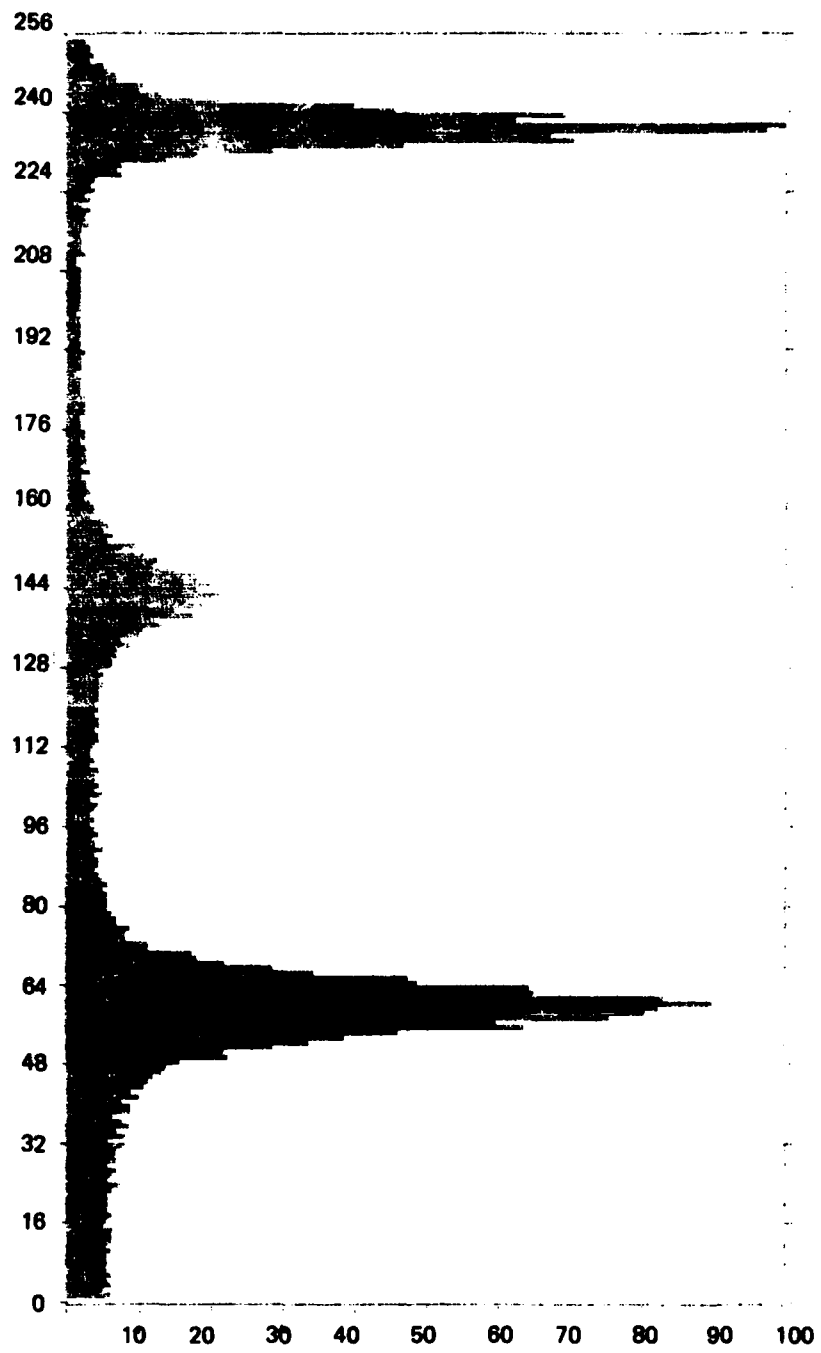


Figure 3. Diffraction pattern of M445 at Zoom II. Channel 60: polyethylene (110) reflection; channel 142.5: polyethylene (200) reflection; channel 237.5: graphite (002) reflection.

Since the area detector method recorded all the data in a pattern simultaneously, only one to three minutes were required to obtain a pattern, compared to a typical data acquisition time of six hours per sample using the older angular scanning method.

Angular calibration was accomplished by measuring the primary beam (suitably attenuated by transmission through copper foils) at intervals of 1.00° on either side of and including the $2\theta = 0.00^\circ$ position in a single pattern. The calibration thus established at the 0 position of the detector arm is transferred to any other range by simply adding the new angular position of the detector arm to the calibration curve. In addition, for increased precision in the determination of d spacings, the polyethylene film samples were coated with a thin layer of graphite (as previously described) to provide an internal reference, the (002) line at $2\theta = 26.62^\circ$.

Finally, to obtain a curve of intensity as a function of 2θ , it is necessary to reduce the two-dimensional data from the detector to a single dimension. However, the diffraction pattern of the randomly oriented polyethylene crystallites appears as a series of arcs on the detector face. In the long run, it would be possible to use all of the information in these arcs by transforming the data from cartesian to angular coordinates. For the present, however, the required intensity curve was obtained by vertically summing the data along a long, narrow strip following the horizontal center line of the pattern. By using only the data on a narrow strip, the effect of the arc curvature may be neglected and the Bragg angle 2θ may be identified with the horizontal cartesian coordinate axis. In the parlance of powder diffractometry, this procedure is equivalent to using a Soller slit system of limited vertical divergence δ as described by Eastabrook.⁵ The effective vertical divergence used in obtaining the present intensity curves was $\delta = 2.2^\circ$ for the "Zoom I" data and $\delta = 1.1^\circ$ for the "Zoom II" data.

The d spacings of the (110) and (200) planes were calculated from the peak diffraction angles using Bragg's well-known equation. The a and b lattice parameters were then calculated using the following equations:

$$a = 2 d_{200} \quad (1)$$

and

$$b^{-2} = d_{110}^{-2} - a^{-2} \quad (2)$$

which are appropriate for the orthorhombic unit cell of polyethylene.

To determine crystallinity from the "Zoom I" data, the intensity curves were mathematically resolved into separate polyethylene amorphous, (110), and (200), and graphite (002) components.⁶ The calculation of crystallinity X_C from the relative areas of the Gaussian polyethylene components followed the method of Gopalan and Mandelkern:⁷

$$X_C = (A_{110} + A_{200}) / (A_{110} + A_{200} + A_{\text{amorph}}) \quad (3)$$

5. EASTABROOK, J. N. *Effect of Vertical Divergence on the Displacement and Breadth of X-Ray Powder Diffraction Lines*. Brit. J. Appl. Phys., v. 3, 1952, p. 349.
6. DESPER, C. R. *Computer Programs for Reduction of X-Ray Diffraction Data for Oriented Polycrystalline Specimens*. Army Materials and Mechanics Research Center, AMMRC TR 72-34, November 1972.
7. GOPALAN, M. R., and MANDELKERN, L. *Degree of Crystallinity of Linear Polyethylene From Wide-Angle X-Ray Diffraction*. Polymer Letters, v. 5, 1967, p. 925.

Use of this equation, however, ignores the effect of the clear lacquer and graphite coating on the specimen surface. This point will be discussed later in greater detail.

Density Determination

Densities were determined in a density gradient column made from water and isopropanol at 23°, using glass beads of known density for calibration. To obtain crystallinity values, limiting values of amorphous and crystalline specific volumes V_A and V_C of 1.170 and 0.999 cm³/g were used based on the data of Gopalan and Mandelkern.⁷ The resulting formula for crystallinity X_C is:

$$X_C = (V_A - 1/\rho)/(V_A - V_C). \quad (4)$$

GPC Determinations

Molecular weight characterization was accomplished using the Waters Model 150-C Gel Permeation Chromatograph. The polymer specimens were dissolved in trichlorobenzene (TCB) and run at 135°C. Two chromatograms were obtained for each sample solution. Data were analyzed against known calibration standards using an on-line microprocessor.

SAMPLES

Samples of DF container material which were studied in previously reported work¹ were available for comparative characterization. Using the terminology of the earlier report, these samples were denoted by both their history and nominal thickness in mm. In terms of history, the samples fall into three classes:

1. OLD - sheets stocked from 1973 production runs;
2. NEW - sheets stocked from 1977 production runs; and
3. EXPOSED - two cannisters which were fabricated in 1973, filled with DF liquid from that date to 1981, then drained and rinsed with water.

Samples are also designated by nominal thickness, either 1.5 mm or 6 mm for different parts of the cannister, although the same polymer resin was used in each instance for both thicknesses.

The above samples were fabricated from the same resin grade (Marlex M407MQ, Philips Petroleum Co.), reportedly a copolymer of ethylene and butene-1, the ethylene being the major constituent. Presumably the butene-1 constituent polymerizes to leave ethyl side groups pendent on a largely linear polyethylene molecule. However, this resin grade has recently been superseded by a new grade, Marlex M445, reportedly a copolymer of ethylene and hexene-1 having quite similar properties. For the M445 these short-chain branching deliberately introduced by incorporation of the comonomer would consist of butyl groups rather than ethyl groups. Both resins fall in a general class of polyolefins described as "Linear Low Density Polyethylenes," or LLDPE's. In actual fact, these resins are of medium density, around 0.94 g/cm³, compared to density values around 0.92 for low density polyethylenes (LDPE's) containing extensive long-chain branching, or to values around 0.96 for high density polyethylenes (HDPE's) which are essentially free of branching of either the short- or long-chain type.

A series of standard samples of varying branch content were also available for calibration purposes. The polymer resins were extensively characterized using methods described by Billmeyer^{8,9} and Williams¹⁰ and made available to the polymer science community in the 1960's by the Plastics Department of E. I. DuPont de Nemours & Co., Inc., Wilmington, Delaware. These samples are labeled "University Contact Polyethylene" and are numbered PE-75, -76, -78, -79, -80, -81, -82, and -85. Characterization data is available in the work of Bodily and Wunderlich.¹¹ The last resin of this series, PE-85, is an essentially linear polymer. Two other linear polyethylenes, Marlex 6007 and Chemplex 6109, were also incorporated into the study. All of the standard samples were available as pellets which were compression molded at 170° to films of 0.2-mm thickness.

In this study, the DF container samples, which were available as thick sheets, were prepared for X-ray studies by microtoming a section of 0.2-mm thickness, which was subsequently heat-relaxed by melting briefly on a glass slide resting on a hot plate at 170°. This treatment was necessary to relieve the residual stresses previously reported¹ in these materials.

The samples were further prepared for X-ray diffraction studies by applying a thin coating of graphite to one side. The graphite was applied by first spraying the surface with a clear lacquer (Illinois Bronze, Federal Stock No. 8010-515-2487), then dusting with graphite powder and spreading the powder evenly with a cotton-tipped applicator.

RESULTS AND DISCUSSION

Molecular Weight Determinations

The gel permeation chromatograms for the three resins are shown in Figure 4, and the computed results are given in Table 1. The results for two of the resins, the 1977 version of M407MQ and the replacement grade M445, are remarkably similar in terms of \bar{M}_n and \bar{M}_w . There is some difference in the \bar{M}_z values but probably not enough in relative terms (roughly 350,000 compared to 370,000) to be a cause for concern. Compared to the earlier resin, M407MQ vintage 1973, both resins are somewhat lower in molecular weight in terms of \bar{M}_n and \bar{M}_w . The most significant difference in the data,

Table 1. POLYETHYLENE MOLECULAR WEIGHT CHARACTERIZATION
BY GEL PERMEATION CHROMATOGRAPHY

Designation	Resin	\bar{M}_n	\bar{M}_w	\bar{M}_z	\bar{M}_w/\bar{M}_n
OLD - 6 mm	M407MQ (1973)	29,300	172,300	369,000	5.88
		30,500	174,100	377,000	5.71
NEW - 6 mm	M407MQ (1977)	20,500	156,700	348,000	7.64
		22,100	160,300	351,000	7.25
M445	M445 (1982)	22,200	163,400	370,000	7.36
		23,100	161,700	367,000	7.00

8. BILLMEYER, F. W., Jr. *Characterization of Molecular Weight Distributions in High Polymers*. J. Polym. Sci., C8, 1965, p. 161.
9. BILLMEYER, F. W., Jr. *Testbook of Polymer Science*. Wiley Interscience, New York, Ch. 3, 1971.
10. WILLIAMS, D. J. *Polymer Science and Engineering*. Prentice-Hall, Englewood Cliffs, New Jersey, Ch. 1, 1971.
11. BODILY, D., and WUNDERLICH, B. *Thermodynamics of Crystalline Linear High Polymers. IV. The Effect of Ethyl, Acetate, and Hydroxyl Side Groups on the Properties of Polyethylene*. J. Polym. Sci.: Part A-2, v. 4, 1966, p. 25.

in fact, is the high \bar{M}_n value of 29,900 for the earliest resin, compared to values of 21,300 and 22,700 for the two later resins. This difference is large enough to be of concern in terms of its possible effect on physical properties and long-term storage behavior as a DF container material. The prime concern would be in terms of resistance to environmental stress cracking, since low molecular weight is known, in general, to have an adverse effect. The literature does not, however, contain information of sufficient detail to permit drawing conclusions about the effect of differences in the molecular weight distribution. We are therefore forced to rely on the results of exposure tests for each particular resin grade. However, the close similarity in molecular weight values between the M407MQ 1977 resin and the M445 resin leads us to predict that, barring significant differences in other parameters, these two grades will perform in similar manner.

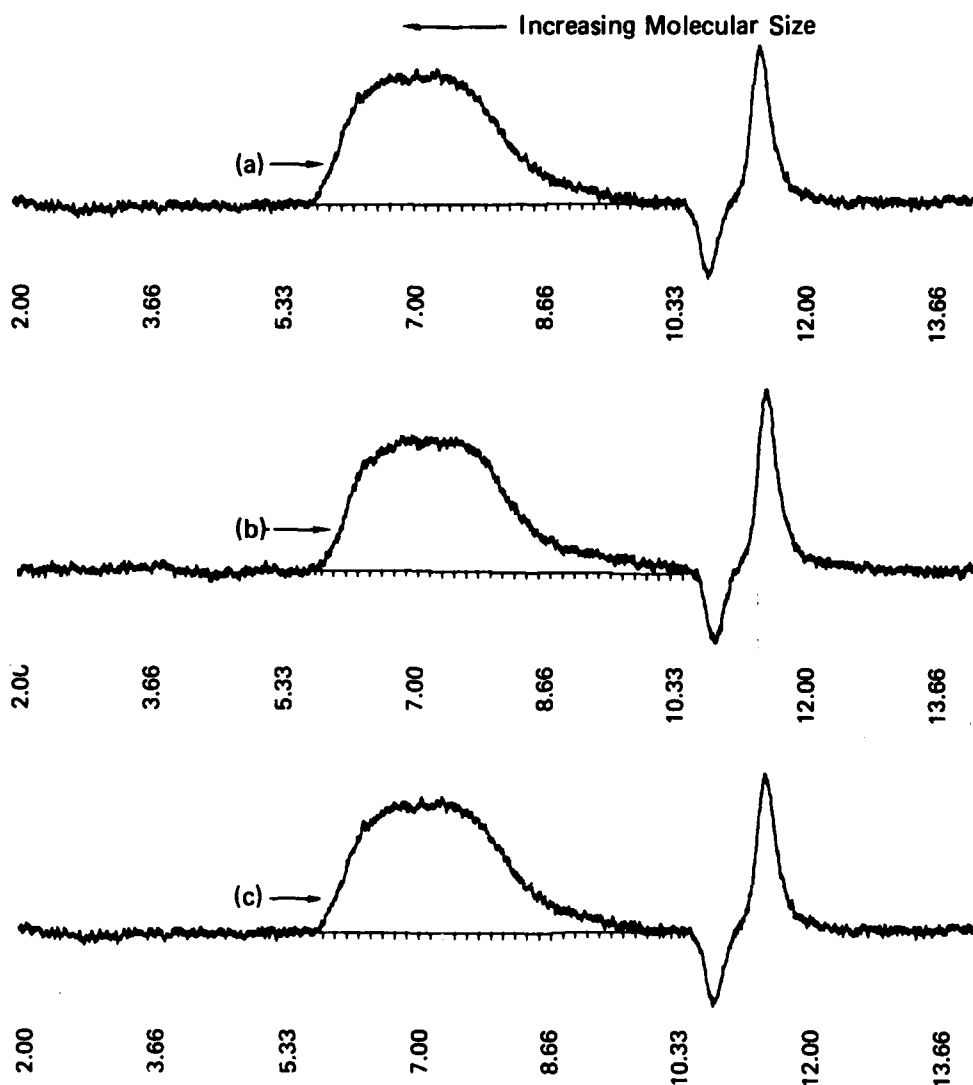


Figure 4. Gel permeation chromatograms: (a) M407MQ (1973); (b) M407MQ (1977); (c) M445.

Characterization of Branch Content

The apparent branch content ($\text{CH}_3/100\text{C}$) from infrared measurements is correlated for the standard samples with the lattice parameters a and b , and with the plane spacing d_{110} in Figure 5. Least squares straight lines drawn through the data yield the equations:

$$(\text{CH}_3/100\text{C}) = 30.17 (a - a_0), \quad (5)$$

$$(\text{CH}_3/100\text{C}) = 103.03 (b - b_0), \text{ and} \quad (6)$$

$$(\text{CH}_3/100\text{C}) = 91.80 (d_{110} - d_{110,0}) \quad (7)$$

where the constants a_0 , b_0 , and $d_{110,0}$ are the intercepts corresponding to the X-ray parameters of a linear polyethylene. The values of the three constants are 7.404, 4.935, and 4.107 Å, respectively.

The X-ray data for the three resins of unknown branch content are used with the correlations of Figure 5 and equations 5 through 7 to calculate their branch contents. The results are given in Table 2.

Data for several of the samples were replicated, allowing an estimate of the precision of the lattice parameter determination. It was found that d_{110} is determined to a standard deviation (σ) of 0.001 Å, while the standard deviation of an a or b determination is 0.004 Å. However, when these values are used in conjunction with the slopes of equations 5 through 7, as summarized in Table 3, the standard deviations of the branch content are found to be nearly equal for the a and d_{110} correlations, but much higher for the b correlation. The key factor is the fact that the a correlation has the lowest slope (equation 5), reflecting the fact that the a dimension of the unit cell expands over three times more rapidly than b with increasing branch content. Thus, although a and b are determined to about the same precision, about three times as much error propagates into the branch content value when

Table 2. BRANCH CONTENT FROM LATTICE PARAMETER MEASUREMENTS

Resin	a , Å	Estd. ($\text{CH}_3/100\text{C}$)	b , Å	Estd. ($\text{CH}_3/100\text{C}$)	d_{110} , Å	Estd. ($\text{CH}_3/100\text{C}$)
M407MQ (1973)	7.432	0.85	4.943	0.78	4.116	0.86
M407MQ (1977)	7.434	0.91	4.946	1.09	4.118	1.04
M445 (1982)	7.432	0.85	4.945	0.99	4.117	0.95

Table 3. PROPAGATION OF ERROR, LATTICE PARAMETER/BRANCH CONTENT CORRELATION

Ordinate	Abscissa	σ (Abscissa)*	Slope of Correlation	σ (Ordinate)*
($\text{CH}_3/100\text{C}$)	a	0.004 Å	30.17%/Å	0.12%
($\text{CH}_3/100\text{C}$)	b	>0.004 Å	103.03%/Å	0.41%
($\text{CH}_3/100\text{C}$)	d_{110}	0.001 Å	91.80%/Å	0.09%

*Standard deviation values are absolute, not relative.

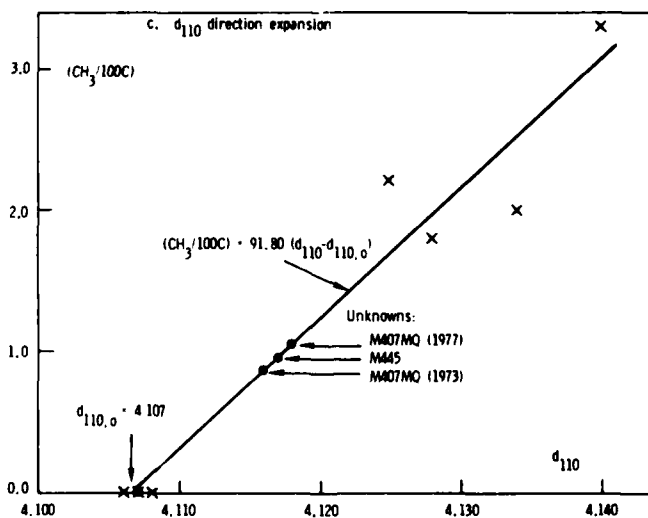
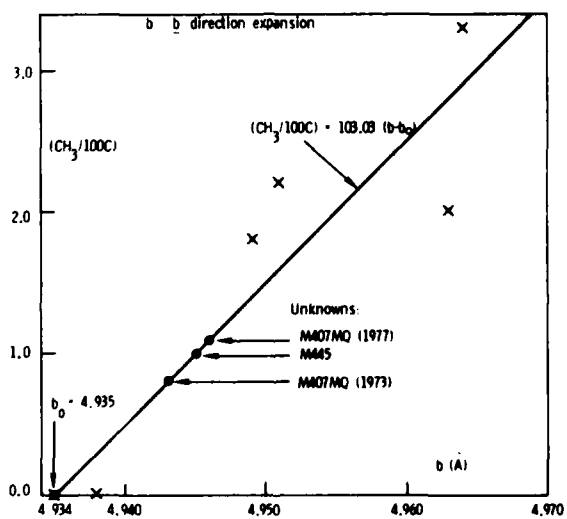
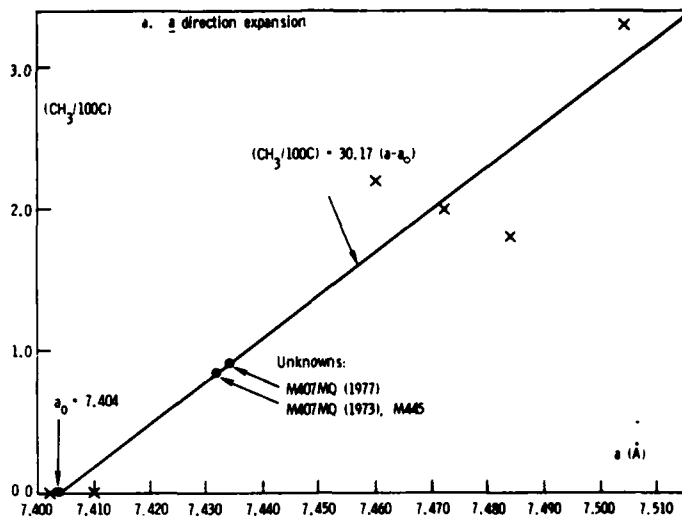


Figure 5. Lattice expansion versus side-chain content in polyethylene.

using the b correlation in place of the a correlation. On the other hand, the parameter d_{110} is also much less sensitive to branch content than is a, but d_{110} is determined to greater precision, since the reflection is the sharpest and most intense of the polyethylene diffraction lines. The two effects tend to cancel out, resulting in comparable precision, on the order of 0.1%, for the branch content when determined from either the a or d_{110} correlations.

At this point it is appropriate to examine the results for the three unknown resins and ask whether there are significant differences between them in branch content. The results, shown in Table 4, are most instructive. Using the a correlation, the average branch content for the three unknown resins is 0.87 with a standard deviation of 0.03 CH₃/100C. However, in Table 3 the estimated precision of such a determination is 0.12 CH₃/100C; in other words, the three unknowns agree with each other to a precision better than the predicted precision of the determination. The same pattern is seen for the results (Table 4) of the b and d_{110} correlations. This could be fortuitous, but a better explanation may be offered by the data in Table 5, in which parameters characterizing the sharpness of the diffraction lines and the signal/noise ratio of the diffraction peaks are listed. The low density resins (PE-75 through PE-80) show broad diffraction lines and low signal/noise ratios, while the linear high-density resins (PE-85, 6007, and 6109) give sharp lines and higher signal/noise ratios. The

Table 4. STANDARD DEVIATIONS* OF BRANCH CONTENT DETERMINATION OF UNKNOWNNS

Ordinate	Abscissa	Average Ordinate	σ (Ordinate)
(CH ₃ /100C)	a	0.87%	0.03%
(CH ₃ /100C)	b	0.95%	0.13%
(CH ₃ /100C)	d_{110}	0.95%	0.07%

*Comparing correlation line results for the three resins of unknown branch content.

Table 5. FACTORS INFLUENCING PRECISION OF DIFFRACTION SPACING MEASUREMENT

Sample	Peak Width at Half Intensity		Peak Signal/Noise Ratio	
	(110)	(200)	(110)	(200)
PE-75	0.582°	0.762°	4.2	1.1
PE-76	0.508	0.716	5.1	1.3
PE-79	0.528	0.722	5.2	1.2
PE-80	0.522	0.704	5.9	2.7
PE-85	0.338	0.390	23.2	4.5
6007	0.334	0.382	17.5	5.7
6109	0.302	0.384	15.1	3.8
M407MQ:				
(1973)	0.408	0.460	10.8	4.1
(1977)	0.390	0.434	14.3	7.3
M445	0.402	0.440	13.7	4.6

three medium density unknowns fall in between, but closer to the high density values of line breadth and signal/noise ratio. The precision of a lattice parameter measurement will be influenced by both factors, with smaller line breadth and higher signal/noise ratio favoring greater precision in a lattice parameter value and in the resulting branch content value. Thus, the precision of a determination is not uniform over the entire range of branch content values. Low density resins will give less precise results, while medium and high density resins will give more precise results. The excellent agreement in Table 4 for the results on the three unknowns is not fortuitous, but reflects the greater precision inherent in the method at lower branch content values.

The lattice parameter method for determining branch content thus becomes more precise in the range where the infrared method (ASTM D2238-68) becomes less precise. For comparison, the relative precision of the lattice parameter correlation is 4% at 3 (CH₃/100C) and 3% at 0.87 (CH₃/100C), while the ASTM standard for the infrared method gives an estimated precision of 2% at 3 (CH₃/100C) and 5% at 0.3 (CH₃/100C).

The accuracy of the two methods must be considered as well as the precision. Precision refers to the reproducibility of a measurement and, generally, takes into account random errors in the measurement. Accuracy refers to the error between the determined result and the true value, and thus includes systematic error as well. There evidently can be a great deal of systematic error in the infrared method, since, although good precision is obtained within a single laboratory, the ASTM standard reports a coefficient of variability of 13% to 30% when results from different laboratories are compared.

The measurement of polyethylene lattice parameters by X-ray diffraction is also subject to the possibility of systematic error arising from errors in instrument alignment. For example, Table 6 lists literature values for the a parameter of linear polyethylene which range from 7.385 to 7.45. In the present work, however, steps have been taken to eliminate the effect of such systematic error on the branch content determination. First, the use of the position-sensitive detector, which records data for all angle values simultaneously rather than sequentially, means that any drift in the instrumentation affects the entire pattern rather than skewing a portion of the pattern. Second, the use of the graphite internal standard diffraction line minimizes the effect of instrumental variables between determinations for different samples. Third, the use of standard polyethylene samples run under the same conditions as the unknowns means that absolute accuracy is not required in the determination of lattice parameters. The diffraction spacings measured here could be in error in absolute terms, but the correlation with branch content is still valid to the precision of the experiment.

The most significant area for improvement in the method is in the standard samples. A wider range of branch content values in the standard samples, as well as more accurately known branch contents would be valuable for improving the correlation coefficient of the least squares fit and for checking for curvature in the correlation.

Crystallinity

The crystallinity values for the three unknowns and for the standard samples, as determined by density and by X-ray diffraction, are listed in Table 7. Serious discrepancies are evident between the density and X-ray crystallinities, and are believed to have their origin in the graphite and lacquer coating applied to the surface to furnish an internal reference line for precise measurement of lattice parameters.

Table 6. LITERATURE VALUES, A PARAMETER OF LINEAR POLYETHYLENE

Author	Date	a
Walter & Reding ¹²	1956	7.38 ₅
Cole & Holmes ¹³	1960	7.40
Swan ¹⁴	1962	7.41 ₇
Wunderlich & Poland ¹⁵	1963	7.40, 7.45
Bodily & Wunderlich ¹¹	1966	7.44 ₂
Zugenmaier & Cantow ¹⁶	1969	7.41 ₈
Kavesh & Schultz ¹⁷	1970	7.38 ₈
Wunderlich ¹⁸	1973	7.42 ₆
This Work	1983	7.40 ₄

Table 7. CRYSTALLINITY VALUES BY DENSITY AND X-RAY DIFFRACTION

Sample	Density ρ (g/cm ³)	Crystallinity, χ_c	
		From ρ	X-Ray*
M407MQ:			
(1973)	0.939 ₅	0.61 ₈	0.50
(1977)	0.942 ₀	0.62 ₄	0.54
M445	0.940 ₉	0.62 ₇	0.55
PE-75	0.914 ₃	0.44 ₆	0.37
PE-76	0.921 ₁	0.49 ₃	0.40
PE-78	0.920 ₃	0.48 ₈	0.44
PE-79	0.921 ₉	0.49 ₉	0.39
PE-80	0.921 ₃	0.49 ₅	0.42
PE-81	0.918 ₉	0.47 ₈	0.40
PE-82	0.917 ₁	0.46 ₆	0.42
PE-85	0.951 ₂	0.69 ₄	0.69
6007	0.953 ₂	0.70 ₇	0.77
6109	0.962 ₀	0.76 ₃	0.60

*Specimens coated with clear lacquer and graphite. Density values of χ_c are preferred.

12. WALTER, E. R., and REDING, F. P. *Variations in Unit Cell Dimensions in Polyethylene*. J. Polym. Sci., v. 21, 1956, p. 561.
13. COLE, E. A., and HOLMES, D. R. *Crystal Lattice Parameters and the Thermal Expansion of Linear Paraffin Hydrocarbons, Including Polyethylenes*. J. Polym. Sci., v. 46, 1960, p. 245.
14. SWAN, P. R. *Polyethylene Unit Cell Variations with Branching*. J. Polym. Sci., v. 56, 1962, p. 409.
15. WUNDERLICH, B., and POLAND, D. *Thermodynamics of Crystalline Linear High Polymers. II. The Influence of Copolymer Units on the Thermodynamic Properties of Polyethylene*. J. Polym. Sci. A, v. 1, 1963, p. 357.
16. ZUGENMAIER, P., and CANTOW, H. J. *Indication of X-Ray Wide Angle Reflexes of Substances of Orthorhombic Unit Cells. Measurements on Polyethylene and Ethylene-Propylene Copolymers*. Kolloid-Z. Z. Polym., v. 230, 1969, p. 229.
17. KAVESH, S., and SCHULTZ, J. M. *Lamellar and Interlamellar Structure in Melt-Crystallized Polyethylene. I. Degree of Crystallinity, Atomic Positions, Particle Size, and Lattice Disorder of the First and Second Kinds*. J. Polym. Sci.: Part A-2, v. 8, 1970, p. 243.
18. WUNDERLICH, B. *Macromolecular Physics*. Academic Press, New York, v. 1, 1973, p. 1-3-154.

This coating evidently contributes diffuse intensity (in addition to the sharp graphite line, whose intensity is accounted for) in the region of the amorphous polyethylene maximum, invalidating the X-ray crystallinity values by making them generally lower than the true crystallinities.

For comparison, Table 8 lists crystallinity values determined by both the density and X-ray methods for six DF container samples previously studied.¹ The two sets of crystallinities are essentially in agreement, even though the X-ray method suffers from greater scatter. The average crystallinity of the six samples is 0.627 ± 0.01 by density, and 0.62 ± 0.03 by X-ray diffraction. The scatter in the X-ray crystallinities is believed to lie in errors associated with: (a) separation of the broad, low amorphous intensity curve from baseline intensity, and (b) separation of the (110) and (200) diffraction peaks from the amorphous peak. Obviously, the X-ray method, based on the calibration of Govalan and Mandelkern⁷ is valid, but the density method of determining crystallinity is preferred.

Table 8. COMPARISON OF DENSITY AND X-RAY VALUES OF CRYSTALLINITY, DF CONTAINER SPECIMENS

Sample	Density ρ (g/cm ³)	Crystallinity, X_c	
		From ρ	X-Ray*
OLD - 1.5 mm	0.939 ₆	0.618	0.60
OLD - 6 mm	0.9394	0.617	0.67
NEW - 1.5 mm	0.9411	0.628	0.63
NEW - 6 mm	0.9430	0.641	0.59
EXPOSED - 1.5 mm	0.9420	0.634	0.62
EXPOSED - 6 mm	0.9402	0.622	0.58
Average	0.9409	0.627	0.62
Standard Deviation	0.0013	0.01	0.03

*Reflection method, no coating.

The density values listed in Tables 7 and 8 for the 1973 and 1977 vintages samples of container material, and for the replacement resin M445, all lie within the range 0.939 to 0.943 g/cm³ given in the specification MIL-P-51431 (EA) for these container resins. In fact, the small differences observed between the six samples of Table 8 may not be particularly significant compared to experimental uncertainty. The six vary by a standard deviation of 0.0013 g/cm³, while replicate determinations of different specimens of a single independent sample yielded a standard deviation of 0.0006 g/cm³. Thus the difference between the six samples of Table 8 is only twice the experimental uncertainty. For all practical purposes, the six container samples of Table 8, and the replacement resin M445, have identical density values.

CONCLUSIONS

The three resin grades studied - M407MQ vintage 1973, M407MQ vintage 1977, and M445 vintage 1982 - proved to be remarkably similar materials in terms of molecular weight and branch (comonomer) content. Although this is evidence of product consistency on the part of the vendor, a clear need remains for close monitoring of materials parameters to meet the requirements for DF containers. The determination

of molecular weight distribution by gel permeation chromatography is a far better guide than the melt flow index and is recommended for inclusion in a materials specification. Precision determination of lattice parameters appears to offer the possibility for more accurate measurement of side chain content, particularly at the level of approximately 1 CH₃/100C, or 0.5% comonomer content, which is of interest for the ethylene/butene-1 and ethylene/hexene-1 copolymers in the LLDPE class of polyolefins. The X-ray lattice parameter method has the potential for better characterization of these materials for branch content than the presently used infrared method. However, characterization of crystallinity by the established density method is the best choice for this purpose.

Once these three parameters (molecular weight distribution, branch contents, and density) are established, the properties of the polyolefin material (aside from processing effects) are fairly well defined. The question of long-chain branching has not been addressed here since this parameter is difficult to assess experimentally. However, the LLDPE class of polyolefins is generally believed to be essentially believed to be essentially free of long-chain branching, as are HDPE's, by the nature of the polymerization reactions used. Only LDPE's, polymerized by the high-pressure free radical mechanism are believed to contain significant amounts of long-chain branches, which have strong effects on the physical, mechanical, and rheological properties. The density specification, however, is judged to cover any possibility of long-chain branching. A polyethylene with a significant level of long-chain branch content will always have a density considerably lower than the specification range of 0.939 g/cm³ to 0.943 g/cm³.

In terms of resin performance, characterization of the three resins indicates that all are in the range of molecular weight, density (crystallinity), and branch content for optimum resistance to environmental stress cracking. The two later resins, M407MQ (1977) and M445, are somewhat lower in number average molecular weight \bar{M}_n than the original M407MQ of 1973. Whether this will have a significant effect on environmental stress cracking in DF containers is best determined by exposure tests which are in progress at the Chemical Systems Laboratory.

ACKNOWLEDGMENTS

The author wishes to gratefully acknowledge the contributions of Dr. Gary L. Hagnauer and Mr. David A. Dunn, who provided the GPC data, and Ms. Judy Brodtkin, who provided the density data.

APPENDIX

1202808 ID NO. - E18112102808
KINETICS OF ENVIRONMENTAL STRESS CRACKING IN HIGH DENSITY POLYETHYLENE.

Bubeck, R. A.

Dow Chem Co, Midland, Mich
 Polymer v 22 n 5 May 1981 p 682-686 CODEN: POLMAG
 ISSN 0032-3861

The observation of environmental stress crack (ESC) growth in high density polyethylene (HDPE) in a 10% Igepal CO-630 solution is reported using double-edge-notched specimens, which allow a fracture mechanics approach. Below the initial stress intensity factor K_{I1} value of 0.4 MPa $m^{1/2}$, the cracking process consisted of both an incubation time for cracking, t/d , and a crack growth stage. The incubation time is stress dependent (decreasing with increasing stress), while the crack growth exhibits a root time ($t \propto \sigma^{-2}$) dependence and is relatively stress independent. The incubation time is the time necessary to generate a dry void craze structure sufficient to allow the PE to absorb the aggressive liquid. The incubation times were determined to be more significant than the actual average crack growth rates for the PE samples tested. Injection molding orientation increases the average crack growth rate without significantly changing the incubation time. 23 refs.

DESCRIPTORS: (*POLYETHYLENES, *Reactions), (CORROSION, Stress Corrosion Cracking), (CHEMICAL REACTIONS, Reaction Kinetics), (POLYMERS, Crack Propagation), SOLUTIONS, MATHEMATICAL TECHNIQUES.

CARD ALERT: 421, 539, 802, 804, 815, 921

1185068 ID NO. - E1811085068
STUDIES OF ENVIRONMENTAL STRESS-CRACK PROPAGATION IN LOW-DENSITY POLYETHYLENE.

Bandyopadhyay, S.; Brown, H. R.

Aust Dep of Health, Abbotsford, Victoria
 J Polym Sci Polym Phys Ed v 19 n 5 May 1981 p 749-761
 CODEN: JPLPAY
 ISSN 0449-2978

In the reported experiments, correlations of the stress-intensity factor K_{I1} with crack speed \dot{a} have been obtained for environmental stress cracking (ESC) of a series of low-density polyethylenes in detergent. In the majority of the materials, the crack speed increases initially with increasing K_{I1} , then becomes constant, and finally starts decreasing. The ESC resistance increases with increasing molecular weight and, in general, the quenched materials show greater ESC resistance than slowly cooled ones. The crack propagation results agree well with the ESC model of Williams. Attempts have also been made to understand the micromechanics of ESC failure from a combined approach of K_{I1} , the crack tip characteristics, and the fracture surface appearance. The roughness of the fracture surface increases with increasing K_{I1} . 17 refs.

DESCRIPTORS: (*POLYETHYLENES, *Crack Propagation), (CORROSION, Stress Corrosion Cracking), DETERGENTS, MATERIALS TESTING

IDENTIFIERS: ENVIRONMENTAL STRESS CRACKING
 CARD ALERT: 421, 802, 804, 815

1184437 ID NO. - E1810868437

CRAZING AND SHEAR LIP FORMATION IN THE NOTCHED CRACK PROPAGATION OF A HIGH DENSITY POLYETHYLENE IN AIR.

Bandyopadhyay, S.; Brown, H. R.

Monash Univ, Clayton, Victoria, Aust
 Mech Behav of Mater, Proc of the Int Conf, 3rd (ICM3), v 3, Cambridge, Engl, Aug 20-24 1979 Publ by Pergamon Press (Int Ser on the Strength and Fract of Mater and Struct), Oxford, Engl and Elmsford, NY, 1980 p 363-371

High density polyethylenes (HDPE) are normally ductile materials and in unnotched tensile tests in air fail by necking and cold drawing, although in service they frequently fail in a brittle manner. Naturally, brittle failure of such a normally ductile material is of concern from a design point of view. In the presence of certain environments, polyethylenes fall in an apparently brittle manner, and the phenomenon, known as environmental stress cracking (ESC) has been widely investigated. 23 refs.

DESCRIPTORS: (*POLYETHYLENES, *Fracture), (MATERIALS TESTING, Tensile Tests)

IDENTIFIERS: CRAZING, MICROGRAPHS
 CARD ALERT: 815, 423

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Polyethylene
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Watertown, Massachusetts 02172-0001
CHARACTERIZATION AND SELECTION OF POLYMER
MATERIALS FOR BINARY MUNITIONS STORAGE -
PART 2: CHARACTERIZATION OF LLDPE RESINS -
C. Richard Desper

Technical Report AMMRC TR 84-28, July 1984, 38 pp -
illus-tables, D/A Project IL162622A554,
AMCMS Code 6126225540A11

This work is aimed at determining the materials properties required for storage of DF (methylphosphonic difluoride) over extended periods of time at temperatures as high as 70°C. Earlier studies showed the most likely mode of failure to be environmental stress cracking of the polyolefine container, rather than chemical or oxidative attack. The most promising candidates in terms of resistance to environmental stress cracking are ethylene copolymers with a small alpha-olefine comonomer content. The comonomer, typically butene-1 or hexene-1, introduces short side chain branches which disrupt the regularity of the crystal morphology, presumably leading to a greater proportion of intercrystalline ties. An X-ray diffraction method was developed for measuring the amount of short chain branching which offers greater accuracy than the previous infrared absorption method. Characterization of short chain branching by such a method, along with molecular weight characterization by gel permeation chromatography and crystallinity determination by density, were deemed essential to quality control of resins for the intended application.

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1159775 ID NO. - E1810759775
ENVIRONMENTAL STRESS CRACKING OF LOW MOLECULAR WEIGHT HIGH DENSITY POLYETHYLENE.
 Bandyopadhyay, S.; Brown, H. R.
 Monash Univ, Clayton, Victoria, Aust
 Polymer v 22 n 2 Feb 1981 p 245-249 CODEN: POLMAG
 ISSN 0032-3861

In the reported study, the stress intensity factor (K) SEM DASH δ crack speed (a) relationships have been obtained for environmental stress cracking (ESC) of specimens of a high density polyethylene having different thermal treatments. Also, scanning electron microscope examination of ESC fracture surfaces has been carried out and correlation between K and the fracture surface appearance has been established. It appears that at low values of K the failure takes place by interlamellar crack propagation; as K increases the mechanism undergoes a transition to void formation and growth, with voids beginning to appear within the spherulites. At high K the failure is entirely by void formation and growth. 15 refs.

DESCRIPTORS: (*POLYETHYLENES, *CORROSION), (CORROSION, Stress Corrosion Cracking), HEAT TREATMENT, MICROSCOPES, ELECTRON, FAILURE ANALYSIS, (MATERIALS TESTING, Fracture).
 CARD ALERT: 408, 421, 537, 741, 802, 815

1148874 ID NO. - E1810648874
ENVIRONMENTAL DYNAMIC FATIGUE CRACK PROPAGATION IN HIGH DENSITY POLYETHYLENE: AN EMPIRICAL MODELLING APPROACH.
 El-Hakeem, H. M.; Culver, L. E.
 Int Coll of Sci & Technol, London, Engl
 Int J Fatigue v 3 n 1 Jan 1981 p 3-8 CODEN: IJFADB
 ISSN 0142-1123

Part of a program to study environmental dynamic fatigue crack propagation in engineering thermoplastics is presented. High density polyethylene has been studied in terms of stress cracking properties under dynamic loading conditions in detergent. The stress cracking was found to vary according to loading conditions and a dependence of crack growth rate on test frequency, amplitude and level of stress intensity factor is reported. An empirical model describing environmental fatigue crack propagation is proposed which adequately represents the experimental results of high density polyethylene. Nylon 66 and which, it is suggested, may be suitable for use with other polymers. 31 refs.

DESCRIPTORS: (*FRACTURE MECHANICS, *Mathematical Models), FATIGUE OF MATERIALS, THERMOPLASTICS, POLYETHYLENES.
 CARD ALERT: 931, 921, 815, 817

Technion, Isr Inst of Technol, Haifa
 Mod Plast v 57 n 11 Nov 1980 p 68-69 CODEN: MOPLAY
 ISSN 0026-8275

Data are presented which show that peroxide-crosslinked HDPE pipe withstands the combination of high temperatures and pressures for long periods of time in hot-water installations. Outdoor applications are now feasible by protection against ultraviolet light. These new types of crosslinked PE pipe resist stress cracking, temperature, pressure, and oxidation. Data are tabulated, plotted, and evaluated. 7 refs.

DESCRIPTORS: (*POLYETHYLENES, *Crosslinking), (CATALYSTS, Peroxides), (PIPE, PLASTIC, Physical Properties), ULTRAVIOLET RADIATION, (CORROSION, Stress Corrosion Cracking), (MATERIALS TESTING, Weathering).
 CARD ALERT: 421, 619, 802, 804, 817, 931

1106671 ID NO. - E1810106671
ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE: ANALYSIS OF THE THREE ZONES OF BEHAVIOR AND DETERMINATION OF CRACK-FRONT DIMENSIONS.
 Shanahan, M. E. R.; Schultz, J.
 CNRS, Mulhouse, Fr
 J Polym Sci Polym Phys Ed v 18 n 8 Aug 1980 p 1747-1752
 CODEN: JPLPAY
 ISSN 0449-2978

Previous work has shown that in the environmental stress cracking (ESC) of polyethylene, there are three major zones of behavior depending on the applied stress and the nature of the liquid environment. These three zones correspond to \$left double quote\$ pure \$right double quote\$ ESC (zone 1), ESC controlled by the speed of penetration of the liquid within a growing crack (zone 2), and behavior as in the absence of liquid (zone 3). Analysis of the transitions between zones has shown that, in the present case, a given liquid will either be capable of giving rise to all three types of behavior depending on the stress applied, or will be totally inactive. A related analysis has enabled the order of magnitude of the dimensions of the crack tip to be estimated and this has been found to be in the range of a micron. 7 refs.

DESCRIPTORS: (*POLYETHYLENES, *Weathering), (CORROSION, Stress Corrosion Cracking), LIQUIDS, MATHEMATICAL TECHNIQUES.
 CARD ALERT: 421, 802, 815, 921, 931

1106555 ID NO. - E1810106555
PIN INDENTATION TEST ON HDPE AND ABS SEM DASHES CIRCUMFERENTIAL STRESS, RETURN SHRINKAGE AND TEST DURATION.
 Orthmann, H. J.
 Kunstst Ger Plast v 70 n 5 May 1980 p 17-19 CODEN: KSGPAT
 This paper shows how the pin or ball indentation test is a proven method for investigating stress cracking in thermoplastics. This test method was used to determine circumferential stress and return shrinkage of HDPE and ABS as a function of test duration. The results show that for durations of up to 100 days and pin diameters of up to 4.6 mm (hole diameter: 3 mm), HDPE may be regarded as predominantly reversibly deformable, ABS as predominantly irreversibly deformable. For figures see German text. 2 refs.
 DESCRIPTORS: (*PLASTICS PRODUCTS, *Physical Properties), (POLYETHYLENES, Molding), (ABS RESINS, Stresses), MATERIALS TESTING.
 IDENTIFIERS: PIN INDENTATION TESTS
 CARD ALERT: 421, 408, 816, 817, 931

1106426 ID NO. - E1810106426
POLYETHYLENE COATED PIPES FOR SUBMARINE PIPELINE.
 Tanaka, M.; Otsuki, F.; Hirano, F.; Sato, T.
 Nippon Steel Corp, Jpn
 Proc Int Conf Intern External Prot Pipes 3rd, v 1, Imp Coll, London, Engl, Sep 5-7 1979. Publ by BHRA (Br Hydromech Res Assoc) Fluid Eng, Cranfield, Bedford, Engl, 1979 p 107-120
 CODEN: PIPPD6
 Polyethylene coated pipes, concrete jacket pipes and non-concrete jacket pipes were developed and their properties were individually investigated. Firstly voids formation between pipe surface and polyethylene and the surface structure of coated polyethylene concerned with the coating process is discussed. Secondly, the mechanism of grip strength of concrete jacket to polyethylene coated pipe is elucidated with polyethylene coated pipes processed by an annular die and a flat die. Thirdly, the importance of adhesive between polyethylene and pipe is made clear in a wide temperature range. Finally effects of surface structure, damages and degradation to environmental stress cracking resistance were investigated in detail with various kinds of polyethylenes. 6 refs.
 DESCRIPTORS: (*PIPELINES, *Offshore).
 CARD ALERT: 619, 512

1092975 ID NO. - E1801292975
DIRECT EVIDENCE FOR THE EXISTENCE OF A CRAZE AT THE CRACK TIP IN ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE.
 Bandyopadhyay, S.; Brown, H. R.
 Monash Univ, Clayton, Victoria, Aust
 Polym Eng Sci v 20 n 11 Jul 1980 p 720-724 CODEN: PYESAZ
 ISSN 0032-3888
 In the reported experimental study, liquid nitrogen fracture tests have been carried out to produce direct evidence of the existence of a voided region, the craze, ahead of the crack in environmental stress cracking of polyethylene. Evidence of crazing is presented for both low and high density polyethylenes. Scanning electron microscopic examination was used, and several micrographs of crack/craze region of fractured samples are included. 21 refs.
 DESCRIPTORS: (*POLYETHYLENES, *Defects), (CORROSION, Stress Corrosion Cracking), (PLASTICS, Crack Propagation), (MATERIALS TESTING, Fracture), MICROSCOPIC EXAMINATION.
 IDENTIFIERS: CRAZING
 CARD ALERT: 421, 741, 802, 817

1092988 ID NO. - E1801292988
METHOD OF MEASURING ENVIRONMENTAL STRESS-CRACKING PERFORMANCE OF POLYETHYLENE BOTTLES.
 Cawood, M. J.; Sleeman, T. J. C.
 BP Chem Ltd, Penarth, South Glamorgan, Wales
 Polym Test v 1 n 3 Jul-Sep 1980 p 191-199 CODEN: POTEDZ
 In this paper a method is described which measures the

performance of blow-moulded polyethylene bottles of specific geometry held under constant pressure in the presence of a detergent solution. An electrical resistance method is used to detect the presence of cracks in the bottle wall. This method has been in use for about 6 years and has been found to correlate with performance not only in blow-moulded containers but in other applications of widely differing geometry. A higher degree of reproducibility than that obtainable from standard test methods is claimed. 7 refs.
 DESCRIPTORS: (*POLYETHYLENES, *Weathering), (BOTTLES, Plastics), (CORROSION, Stress Corrosion Cracking), ENVIRONMENTAL IMPACT.
 CARD ALERT: 421, 694, 802, 817, 901

1092975 ID NO. - E1801292975
DIRECT EVIDENCE FOR THE EXISTENCE OF A CRAZE AT THE CRACK TIP IN ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE.
 Bandyopadhyay, S.; Brown, H. R.
 Monash Univ, Clayton, Victoria, Aust
 Polym Eng Sci v 20 n 11 Jul 1980 p 720-724 CODEN: PYESAZ
 ISSN 0032-3888
 In the reported experimental study, liquid nitrogen fracture tests have been carried out to produce direct evidence of the existence of a voided region, the craze, ahead of the crack in environmental stress cracking of polyethylene. Evidence of crazing is presented for both low and high density polyethylenes. Scanning electron microscopic examination was used, and several micrographs of crack/craze region of fractured samples are included. 21 refs.
 DESCRIPTORS: (*POLYETHYLENES, *Defects), (CORROSION, Stress Corrosion Cracking), (PLASTICS, Crack Propagation), (MATERIALS TESTING, Fracture), MICROSCOPIC EXAMINATION.
 IDENTIFIERS: CRAZING
 CARD ALERT: 421, 741, 802, 817

Meyer, C. T.; Filippini, J. C.; Felicit, N.

CNRS, Grenoble, Fr
 Annu Rep Conf Electr Insul Dielectr Phenom Annu Meet 47th, Pocono Manor, Pa, Oct 30-Nov 2 1978. Available from NAS, Washington, DC, 1978 p 374-381 CODEN: CEIPAZ
 After a critical examination of the literature on water treeing in polyethylene insulating and of the proposed mechanisms involving the mechanical characteristics of the polymer, and interpretation based on an analogy between water tree propagation and stress cracking is proposed. A comparison is made between the electrostatic energy and the energy required for the propagation of a crack. Although available data on surface energies lead to very high values of the local electric field, this assumption cannot be rejected. The necessity of further experimental investigations is stressed. 18 refs.
 DESCRIPTORS: (*ELECTRIC INSULATING MATERIALS. *Plastics). (POLYETHYLENES, Crack Propagation).
 IDENTIFIERS: WATER TREEING
 CARD ALERT: 413, 817, 701

1039560 ID NO. - E1800539560

CORRELATION BETWEEN ENVIRONMENTAL STRESS CRACKING AND LIQUID SORPTION FOR LOW-SWELLING LIQUIDS.

Shanahan, M. E. R.; Schultz, Jacques
 Ec Natl Super de Chim de Mulhouse, Fr
 J Polym Sci Polym Phys Ed v 18 n 1 Jan 1980 p 19-26
 CODEN: JPLPAY
 ISSN 0449-2978

In the reported experiments, the environmental stress cracking (ESC) of polyethylene has been studied under conditions of dynamic equilibrium with the liquid for low-swelling liquids. Even among active ESC agents, there is a clear relative order of efficiency. It has been shown that the liquid becomes less efficient with increasing equilibrium swelling. This fact has been attributed to local plasticization of the crack front leading, in turn, to a reduction in the high-stress concentrations associated with a wedge-shaped crack. Some semiquantitative ideas are proposed in an attempt to explain the relation between ESC efficiency and volume/volume sorption. 6 refs.

DESCRIPTORS: (*POLYETHYLENES, *Permeability), (LIQUIDS, Sorption), (CORROSION, Stress Corrosion Cracking), (POLYMERS, Stresses), MATHEMATICAL TECHNIQUES.
 IDENTIFIERS: ENVIRONMENTAL STRESS CRACKING, SWELLING
 CARD ALERT: 408, 539, 802, 815, 921, 931

1092973 ID NO. - E1801292973
 EFFECT OF SURFACTANT SOLUTIONS ON THE GROWTH RATE OF ENVIRONMENTAL STRESS CRACKING OF LOW-DENSITY POLYETHYLENE.

Ohda, Yoshihito; Okamoto, Hiroshi
 Nagoya Inst of Technol, Jpn
 J Mater Sci v 15 n 6 Jun 1980 p 1539-1546 CODEN: JMTSAS
 ISSN 0022-2461

The growth rate of environmental stress cracking (ESC) of low-density polyethylene (PE) specimens bent and immersed in surfactant solutions has been measured. In the temperature range 25 to 60 degrees C, the values of the growth rate are almost independent of the amount of deformation varying in a narrow range but dependent on it below 25 degrees C. They are changed over a few hundredfold by the changes of temperature and of surfactant solutions. the growth of ESC of PE is assumed to be a stress-assisted thermal activation process. In the temperature range 25 to 60 degrees C, the values of activation enthalpy obtained depend on the surfactants used, but are almost independent of their concentrations. The nature of the mixture-like (surfactant and PE) region developed on PE surfaces at crack tips and the increase in the mobility of PE segments there seem to be essential to ESC of PE. 17 refs.

DESCRIPTORS: (*POLYETHYLENES, *Crack Propagation), ENVIRONMENTAL TESTING, STRESSES, (PLASTICS, Fracture).
 IDENTIFIERS: SURFACTANTS, ENVIRONMENTAL STRESS CRACKING
 CARD ALERT: 817, 421

1092793 ID NO. - E1801292793

QUALITY OF POLYETHYLENE COATING FOR ARCTIC SERVICE PIPELINE.

Tanaka, Mansel; Ayuenwa, Saburo; Ohtsuki, Fuyuhiko; Sugimura, Shigeyuki; Yoshida, Hiroshi
 Nippon Steel Corp, Kimitsu, Jpn
 Nippon Steel Tech Rep Overseas n 14 /379 p 112-119 CODEN: NSTTID

Protective coatings of pipeline for arctic service are required to have excellent qualities over a wide temperature range. Qualities such as low temperature impact resistance, flexibility, stress cracking resistance, and high temperature degradation resistance as the pipe temperature rises to 80 degrees C after the pipeline is put into service. A description is given of large-diameter polyethylene coated pipes with excellent performance for arctic service after study of polyethylene structure, combinations of adhesives, and their effects on coating performance. 11 refs.

DESCRIPTORS: (*PIPELINES, STEEL, *Protective Coatings), (PROTECTIVE COATINGS, Plastics).
 CARD ALERT: 545, 619, 817

1044191 ID NO. - E1800644191

WATER TREE PROPAGATION IN RELATION TO MECHANICAL PROPERTIES OF POLYETHYLENE

1023882 ID NO. - E1800323882
LIFETIME BEHAVIOR OF POLYETHYLENE SHEETS UNDER INFLATION IN THE PRESENCE OF STRESS-CRACKING AGENT.

Crisman, J. M.; Zepas, L. J.
NBS, Washington, DC
Am Chem Soc Div Org Coat Plast Chem Prepr V 41, Pap presented at Natl Meet of the Am Chem Soc, 178th, Washington, DC, Sep 9-14 1979. Publ by Am Chem Soc, Div Org Coat and Plast Chem, Washington, DC, 1979 p 475-480 CODEN: ACOCAO ISSN 0096-512X

From a constitutive equation and the assumption that the strain potential energy function for small deformations does not vary much over the surface covered by the strain invariants $I/1$ and $I/2$, one can predict that an instability should occur in equal biaxial creep at a strain equal to about one half that for which the instability occurs in uniaxial extension. Since during inflation at constant pressure the engineering stress is not constant, the calculation of the instability is much more complex and the exact point of instability can only be estimated. At low pressures where failure occurs by cracking, the stress initially decreases and then either continues to decrease up to the point of fracture, or at the lowest pressures tends to level off to a nearly constant value. 7 refs.

DESCRIPTORS: (*POLYETHYLENES, *Fracture), (PLASTICS FILMS, Fracture).
CARD ALERT: 815, 817

1023880 ID NO. - E1800323880
STATIC FATIGUE OF POLYETHYLENE IN UNIAXIAL CREEP IN THE PRESENCE OF STRESS-CRACKING AGENTS AND SOLVENTS.

Zepas, L. J.; Crisman, J. M.
NBS, Washington, DC
Am Chem Soc Div Org Coat Plast Chem Prepr V 41, Pap presented at Natl Meet of the Am Chem Soc, 178th, Washington, DC, Sep 9-14 1979. Publ by Am Chem Soc, Div Org Coat and Plast Chem, Washington, DC, 1979 p 469-474 CODEN: ACOCAO ISSN 0096-512X

Static fatigue in uniaxial loading for two types of polyethylene. The behavior in air at two different temperatures will be compared to that in solvent or stress-cracking agent at the same two temperatures. The authors have attempted to separate out one failure mechanism arising mainly from bond rupture which ultimately leads to crack propagation which is greatly influenced by the presence of stress-cracking agents. 6 refs.

DESCRIPTORS: (*POLYETHYLENES, *Fatigue).
CARD ALERT: 815

1023873 ID NO. - E1800323873
INFLUENCE OF PROCESSING DEGRADATION ON FINAL PROPERTIES OF POLYETHYLENE SHEATHING COMPOUNDS.

de Bellet, J. J.
Union Carbide Eur, Geneva, Switz
Plast in Telecommun 2, IEE, Int Conf, London, Engl, Sep 18-20 1978 Publ by Plast and Rubber Inst, London, Engl, 1978 p 29, 1-29, 11

Influence of processing degradation, in an internal mixer, on final properties of sheathing polyethylene compounds has been determined through a computerized thermodynamic analysis. Calculation of degradation energy, its effect on environmental stress cracking resistance and other physical properties is also presented. 4 refs.

DESCRIPTORS: *POLYETHYLENES, TELECOMMUNICATION CABLES.
CARD ALERT: 815, 716, 718

937425 ID NO. - E1791297425
FUNDAMENTAL MECHANISMS OF ENVIRONMENTAL STRESS CRACKING IN POLYETHYLENE.

Lustiger, A.; Corneliusen, R. D.
Drexel Univ, Philadelphia, Pa
SPE, Annu Tech Conf, 37th, New Orleans, La, May 7-10 1979 Publ by SPE, Greenwich, Conn, 1979 p 606-609

Experimental results are presented which demonstrate that crack propagation through shear bands and crazes are critically important steps for environmentally induced crack formation in low and high density polyethylene respectively. The action of the surfactant environment on low density polyethylene can be explained by surfactant absorption and subsequent plasticization thus initiating shear bands. In high density polyethylene, however, cracks initiate from voids near the specimen surface which accompany crystallite deformation. The highly polar surfactant induces a spreading pressure in these voids which initiate crazes. 10 refs.

DESCRIPTORS: (*POLYETHYLENES, *Chemical Resistance), (*CORROSION, Stress Corrosion Cracking), (POLYMERS, Crack Propagation), SURFACE ACTIVE AGENTS, CHEMICAL REACTIONS, IDENTIFIERS: ENVIRONMENTAL STRESS CRACKING, REACTION MECHANISMS
CARD ALERT: 421, 802, 803, 804, 815

992844 ID NO. - E1791292844
SPECIFICATION METHOD FOR ULTRA HIGH MOLECULAR WEIGHT POLYETHYLENE FOR IMPLANT USE.
 Wagner, H. L.; Dillon, J. G.
 NBS, Washington, DC
 J Biomed Mater Res v 13 n 5 Sep 1979 p 821-824 CODEN: JBMR8G
 ISSN 0021-9304

Ultra high molecular weight polyethylene has gained wide acceptance as an implant material with excellent resistance to creep, stress cracking and wear. Because many of these desirable properties are a function of its high molecular weight, a reliable specification method of determining molecular weight of the order of 10^{+6} minus 10^{+7} is needed for controlling and optimizing these properties. The purpose of this note is to point out that the estimated molecular weight of ultra high molecular weight polyethylene (UHMWPE) obtained from single point viscosity measurements may be significantly in error when these estimates are derived by methods which are appropriate for lower molecular weight polymers. Single point viscosity measurements, carried out under standardized conditions, should be used only as molecular weight indices, without any quantitative association with absolute molecular weights. The method the authors have employed is outlined below and is similar to the one being proposed by ASTM Committee D 20. 12 as a standard method. 4 refs.

DESCRIPTORS: (*BIOMEDICAL ENGINEERING. *Surgical Implants). (POLYETHYLENES. Molecular Weight). (MATERIALS TESTING. Standards).
 IDENTIFIERS: BIOMATERIALS
 CARD ALERT: 461, 817, 423, 902

981398 ID NO. - E1791081398
EFFECTS OF RADIATION STERILIZATION ON THE PROPERTIES OF ULTRAHIGH MOLECULAR WEIGHT POLYETHYLENE.
 Nusbaum, H. J.; Rose, R. M.
 MIT, Cambridge, Mass
 J Biomed Mater Res v 13 n 4 Jul 1979 p 557-576. CODEN: JBMR8G
 ISSN 0021-9304

The effects of radiation sterilization on ultrahigh molecular weight polyethylene were explored by freeze fractures, electron spin resonance, absorption of aqueous media, density measurements, small-angle and wide-angle x-ray scatter, differential scanning calorimetry, infrared and ultraviolet-visible spectrometry, static and dynamic mechanical properties measurements, mechanical fatigue measurements, and standard environmental stress cracking measurements. The rate of fluid absorption increased significantly due to the appearance of carbonyl groups. As the fluids were absorbed, density increased and small-angle x-ray scatter (SAXS) intensity diminished slightly, as expected. Small changes in mechanical properties (e. g.

tensile properties and dynamic spectrum) were always consistent with the cross-linking which occurs after irradiation; the only significant mechanical deterioration was in the fatigue properties. The nonpherulitic, inhibited crystallinity which characterized this material and which is responsible for its excellent static resistance to environmental stress cracking was not at all changed. 14 refs.

DESCRIPTORS: (*POLYETHYLENES. *Radiation Effects). STERILIZATION, (PLASTICS. Crosslinking). (MATERIALS TESTING. Fatigue).
 IDENTIFIERS: BIOMATERIALS. ULTRA-HIGH-MOLECULAR-WEIGHT POLYETHYLENES
 CARD ALERT: 817, 622, 462, 421, 422

972507 ID NO. - E1790972507
LIFETIME BEHAVIOR OF POLYETHYLENE BARS IN UNIAXIAL EXTENSION IN VARIOUS CHEMICAL ENVIRONMENTS.
 Crisman, J. M.; Zapas, L. J.
 NBS, Washington, DC
 ACS Symp Ser n 95, Durability of Macromol Mater. from a Symp at the Meet of the ACS, 178th, Miami, Fla, Sep 11-14 1978. Publ by ACS, Washington, DC, 1978 p 289-299 CODEN: ACSM8C

In the work described here, fracture data are presented for two types of polyethylene. The behavior in air at two different temperatures is compared to that in solvent or stress-cracking agent at the same two temperatures. More specifically, creep data as a function of time up to the point of fracture were obtained under uniaxial loading for each combination of temperature and environment. It has been shown for two different types of polymers under conditions of dead load fatigue in uniaxial extension that brittle fracture can be represented by a mechanism whose contributions become important at small loads, or very slow creep rates. This mechanism is also very much affected by the presence of stress-cracking agents. It is found that the adverse effect due to stress-cracking agent is much more emphatic than in the case of uniaxial extension. Since stress-cracking tests traditionally have been designated for short-time testing, it becomes clear why the folklore came into being that stress-cracking occurred only under biaxial conditions. In solvents such as hexane or dodecane, the lifetime is shortened even more than in the case in stress-cracking liquid. 8 refs.

DESCRIPTORS: (*POLYETHYLENES. *Chemical Resistance). (MATERIALS TESTING, Tensile Tests). (SOLVENTS. Chemistry). (POLYMERS. Fracture). (CORROSION. Stress Corrosion Cracking).
 CARD ALERT: 421, 802, 804, 815

960655 ID NO. - E1790860855

EVALUATION OF SELECTED PLASTICS FOR PROJECTED OTEC HEAT EXCHANGERS.

Henke, Thomas E.; Hartt, William H.; Hart, Geoffrey K.
 Fla Atl Univ, Boca Raton
 Mater Performance v 18 n 5 May 1979 p 9-17 CODEN: MTPFBI
 ISSN 0094-1492

Accelerated test data are given for high density PE, polypropylene, polybutylene, PVC, acetal copolymer nylon, and cellulose acetate butyrate in air, sea water, liquid ammonia, propane, and isobutane for periods up to 127 days. Reactions are tabulated for weight change, dimension change, tensile properties and appearance. Stress cracking data are given on HDPE, polybutylene, and acetal. Environments were representative of those expected for ocean thermal gradient power systems. 5 refs.
 DESCRIPTORS: (*ELECTRIC POWER PLANTS, *Offshore), POWER GENERATION, (PLASTICS, Applications).
 IDENTIFIERS: OFFSHORE THERMAL ENERGY CONVERSION (OTEC)
 CARD ALERT: 611, 615, 817

945741 ID NO. - E1790645741

ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE: CRITERIA FOR LIQUID EFFICIENCY.

Shenahan, M. E. R.; Schultz, J.
 Ec Natl Super de Chim de Mithouse, Fr
 J Polym Sci Polym Phys Ed v 17 n 4 Apr 1979 p 705-710
 CODEN: JPLPAY
 ISSN 0449-2978

In the reported experiments, the environmental stress cracking (ESC) of polyethylene in nonreacting, nonswelling liquids has been studied using uniaxial creep tests. For active liquids, three types of behavior have been recognized. At low stresses, left double quotes pure right double quotes ESC occurs; at intermediate stresses, time to failure is largely controlled by the ability of the liquid to flow into a growing crack; and at high stresses, ESC is in competition with failure by necking, and the latter prevails. The liquid does not therefore play a significant role in this last case. Nonactive liquids produce results similar to those observed in air. It is believed that this is because these liquids are unable to flow into growing cracks sufficiently quickly even at low stresses and thus the liquid does not influence failure behavior. This criterion for activity of the liquid is largely determined by the viscosity of the liquid and by the spreading coefficient of the liquid on the solid SEM DASH\$ a parameter defining the ability of the liquid to wet the solid. 3 refs.

DESCRIPTORS: (*POLYETHYLENES, *Corrosion), (CORROSION, Stress Corrosion Cracking), LIQUIDS, (MATERIALS TESTING, Creep).
 CARD ALERT: 421, 802, 817, 931

938789 ID NO. - E1790536789

ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE: TEMPERATURE EFFECT.

Soni, P. L.; Gell, P. H.
 Case West Reserve Univ, Cleveland, Ohio
 J Appl Polym Sci v 23 n 4 Feb 15 1978 p 1167-1179 CODEN: JAPNAB
 ISSN 0021-8995

In the reported experiments, polyethylene was drawn at temperatures ranging from 30 degrees to 60 degrees C in aggressive and nonaggressive environments. Fibrillation was found to occur in the aggressive environment, and this effect increased with temperature. The temperature effect was more prominent at lower strain rates. Thin films drawn in aggressive environments deformed inhomogeneously. Again, this effect was found to increase with increasing temperatures. Single crystal deformation was also found to be inhomogeneous, and left double quotes solvation right double quotes of the amorphous surface layer occurred in the presence of the aggressive environment. Infrared measurements of sorption under different loads indicated that there is an increase in the amount of sorbed materials with increasing load. Dynamic mechanical studies revealed the intracrystalline regions to be affected preferentially. 14 refs.

DESCRIPTORS: (*POLYETHYLENES, *Drawing and Stamping), THERMAL EFFECTS, (CORROSION, Stress Corrosion Cracking), (CRYSTALS, Defects), SPECTROSCOPY, INFRARED, CHEMICALS.
 IDENTIFIERS: ENVIRONMENTAL STRESS CRACKING, SINGLE CRYSTAL DEFORMATION
 CARD ALERT: 641, 801, 802, 804, 815

927400 ID NO. - E1790427400
 USE OF C-SHAPED SPECIMEN FOR EVALUATING THE SLOW CRACK
 GROWTH RESISTANCE OF HOLLOW CYLINDRICAL PARTS.

Lee, Chul Soo; Epstein, Michael M.
 Batelle, Columbus Lab, Ohio
 SPE Natl Tech Conf: Plast in Packag and Acrylonitrile-A Dual
 Meet, Chicago, Ill, Nov 13-15 1978 Publ by SPE, Stamford,
 Conn, 1978 p 94-9

In the reported experiments, the slow crack growth test was first performed with standard three point notched-bar bend fracture specimens machined from a 4-inch (101.6 mm) SDR11 PE23061 pipe. The test results were used as a reference to compare similar test results obtained from the C-shaped test specimens prepared from the same material. Later, the C-shaped test specimens were used in evaluating the crack growth resistance of several 2-inch (50 mm) IPS SDR11 piping materials which cover a wide range of environmental stress cracking behavior. It is shown that long-term, slow crack growth resistance of polyethylene pipe samples can successfully be evaluated in a relatively short time by using C-shaped test specimens. Test results compare well with those obtained from standard notched bar specimens. 5 refs.

DESCRIPTORS: (*PIPE, PLASTIC, *Crack Propagation). (MATERIALS TESTING, Fracture). (POLYETHYLENES, Testing). IDENTIFIERS: C-SHAPED TEST SPECIMENS
 CARD ALERT: 421, 619, 817

911854 ID NO. - E1790211854
 THEORY OF THE ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE.

Brown, H. R.
 Monash Univ, Clayton, Victoria, Aust
 Polymer v 19 n 10 Oct 1978 p 1186-1188 CODEN: POLMAG
 It is proposed that the environmental stress cracking of polyethylene is caused by stress-induced swelling and plasticization of certain favorably oriented amorphous regions in polyethylene. The criteria for stress-induced swelling together with the criteria for little swelling at zero stress show that a vigorous stress cracking agent will have a solubility parameter close to that of polyethylene and a large molar volume. Some detergents fit into this category. 27 refs.

DESCRIPTORS: (*POLYETHYLENES, *Stresses). (CORROSION, Stress Corrosion Cracking). MATHEMATICAL TECHNIQUES. CHEMICALS, DETERGENTS, IDENTIFIERS: STRES-INDUCED SWELLING
 CARD ALERT: 408, 802, 804, 815

911836 ID NO. - E1790211836
 SELECTIVE CHEMICAL ETCHING OF POLY(ETHYLENE TEREPHTHALATE) USING PRIMARY AMINES

Sweet, G. E.; Bell, J. P.
 Univ of Conn, Storrs

J Polym Sci Polym Phys Ed v 16 n 11 Nov 1978 p 1935-1946
 CODEN: JPLPAY

In the reported experiments, several primary amines have been examined as selective degradative etchants for the investigation of poly(ethylene terephthalate) (PET) morphology. The objective is to remove less ordered regions, leaving crystals intact. The amines include 40% and 20% aqueous methylethylamine, 70%-40% aqueous ethylamine and pure and 40% aqueous n-propylamine. Weight-loss and x-ray diffraction data show that certain concentrations of aqueous amine solutions simultaneously degrade and crystallize PET. This observation indicates the hazard of using some of these amine reagents to characterize PET morphology since the crystalline structure found after etching is likely to be a result of solvent-induced crystallization during degradation. Data for 40% aqueous methylethylamine used at room temperature shows that crystallization does not occur during etching, and in light of earlier research indicates the favorable nature of this reagent as a selective degradation medium for PET. Applications of this reagent disclosed that in oriented PET fibers chemical stress cracking occurs, causing the degradative reagent to lose its selectivity. 28 refs.

DESCRIPTORS: (*POLYESTERS, *Etching). NITROGEN COMPOUNDS. (CRYSTALS, Structure). X-RAY ANALYSIS, SYNTHETIC FIBERS. (POLYMERS, Degradation). IDENTIFIERS: POLYETHYLENE TEREPHTHALATE. AMINES. MORPHOLOGIES
 CARD ALERT: 801, 802, 804, 815, 819

911835 ID NO. - E1790211835
CHEMICAL DEGRADATIVE STRESS CRACKING OF POLY(ETHYLENE TEREPHTHALATE) FIBERS.

Sweet, G. E.; Bell, J. P.
 Univ of Conn, Storrs
 J Polym Sci Polym Phys Ed v 16 n 11 Nov 1978 p 2057-2077
 CODEN: JPLPAY

Poly(ethylene terephthalate) (PET), after certain thermal and mechanical histories, exhibits stress cracking when exposed to 40% aqueous methyamine. This reagent has also been used for selective degradation of PET films. Stress cracking is shown to occur during degradation only when a specimen supports an internal or externally applied stress above a critical level. The cracking density in a filament is shown by the present work to increase as the draw ratio is increased or when the fiber is highly annealed. This increased cracking is associated with an increase in the magnitude of the internal residual stresses resulting from molecular orientation developed during these processes. Because of this, crack density and fiber birefringence were found to correlate well. In addition, the geometry of the stress-cracking pattern along a filament is affected by internal residual stresses since the propagation of spiral and helical cracks results from the effect of a biaxial stress field remaining at the filament surface after processing. 27 refs

DESCRIPTORS: (*POLYESTERS, *Degradation), (CORROSION, Stress Corrosion Cracking), (SYNTHETIC FIBERS, Stresses).
 CHEMICAL REACTIONS, (CHEMICALS, Chemistry).
 IDENTIFIERS: POLYETHYLENE TEREPHTHALATE, CRACK FORMATION, AQUEOUS METHYLAMINE
 CARD ALERT: 408, 802, 815, 819

892683 ID NO. - E1781292683
LIFETIME BEHAVIOR OF POLYETHYLENE BARS IN UNIAXIAL EXTENSION IN VARIOUS CHEMICAL ENVIRONMENTS.

Crisman, J. M.; Zepes, L. J.
 NBS, Polym Sci & Stand Div, Washington, DC
 Am Chem Soc Div Polym Chem Prepr v 19 n 2, Pap presented at the Natl Meet, 176th, Miami Beach, Fla, Sep 9-15 1978. Publ by Am Chem Soc, Div Polym Chem, Washington, DC, Sep 1978 p 799-804 CODEN: ACPPAY

In the study of the mechanical properties of amorphous materials the use of elevated temperature and solvents has provided one relatively successful means for obtaining long time behavior, i. e., long relaxation times, from short time experiments. Provided the proper mechanisms can be specified, the same procedure should be applicable to semicrystalline polymers in cases where long times are required to fracture the material. In the work described, fracture data are presented for two types of polyethylene. The behavior in air at two different temperatures is compared to that in solvent or stress-cracking agent at the same two temperatures. More specifically, creep data as a function of time up to the point

of fracture were obtained under uniaxial loading for each combination of temperature and environment. It is demonstrated that it is possible to use either a solvent or a surface active type of stress-cracking agent as a means of accelerating fracture. In the case of 1630 in water, the authors have applied a superposition principle in order to separate out one mechanism, that arising from bond rupture and crack formation. A proper description of this mechanism should be valuable as a means of predicting long time behavior under conditions where the applied load may be very small. 4 refs.

DESCRIPTORS: (*POLYETHYLENES, *Creep), (MATERIALS TESTING, Tensile Tests), (POLYMERS, Corrosion), THERMAL EFFECTS, SOLVENTS.
 CARD ALERT: 421, 539, 804, 815

860302 ID NO. - E1780860302
ENVIRONMENTAL STRESS CRACKING AND MORPHOLOGY OF POLYETHYLENE.

Bandyopadhyay, S.; Brown, H. R.
 Monash Univ, Clayton, Victoria, Aust
 Polymer v 19 n 5 May 1978 p 589-592 CODEN: POLMAG
 In the reported experiments, studies have been made of the fracture surfaces of high density polyethylene which failed by environmental stress cracking at very low stresses. The failure appears to be almost entirely brittle, contrary to the evidence of plastic deformation and void formation reported by other workers. The failure occurred either in an interlamellar manner within a spherulite, or, when the lamellae matched poorly across an interspherulitic boundary, by cracking along that boundary. Because of the brittle nature of the failure, the fracture surface provides information on spherulite morphology in bulk crystallized material. 9 refs.

DESCRIPTORS: (*POLYETHYLENES, *Microstructure), (Stress Corrosion Cracking), (POLYMERS, Fracture), IDENTIFIERS: MORPHOLOGIES
 CARD ALERT: 421, 801, 802, 815

Lustiger, A.; Corneliussen, R. D.; Kantz, M. R.
Drexel Univ., Philadelphia, Pa

Mater Sci Eng v 33 n 1 Apr 1978 p 117-123 CODEN: MSCEAA
Stages of environmental stress cracking in low-density polyethylene were studied using scanning electron microscopy. Environmental stress cracking was induced in a surfactant bath using two well established methods. It was found that both crazes and shear bands appear on specimen surfaces during the early stage of stress cracking. Elliptical cracks subsequently grow out of the shear bands and ultimately connect to form macroscopic cracks. In addition to the appearance of the elliptical cracks, subsurface cracks formed in specimens subjected to bending. Fracture initiates when the subsurface cracks ultimately join with the surface cracks generated from shear bands. 20 refs.

DESCRIPTORS: (*POLYETHYLENES, *Crack Propagation), (ENVIRONMENTAL TESTING, Stresses), IDENTIFIERS: ENVIRONMENTAL STRESS CRACKING
CARD ALERT: 815, 817, 421, 423

852468 ID NO. - E1780752468 DETERGENT STRESS-CRACKING OF POLYETHYLENE.

Owen, D. Roger J.; Haward, Robert N.; Burbury, Anthony
Univ of Swansea, Wales
Br Polym J v 10 n 1 Mar 1978 p 98-102 CODEN: BPOJAB

Description of the results obtained for model calculation on the failure of an elastic-plastic solid with properties approximating to those of different types of polythene at 50C, the temperature used for the conventional detergent stress-cracking test. Experimental and electron microscopic evidence supporting the conclusion reached from the model is also presented. 27 refs.

DESCRIPTORS: (*POLYETHYLENES, *Mechanical Properties),
CARD ALERT: 815

852478 ID NO. - E1780752478 INFLUENCE OF WETTING PROPERTIES OF THE LIQUID ON ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE AT HIGH STRESS.

Shanahan, M. E. R.; Schultz, J.
Univ du Haut-Rhin, Mulhouse, Fr
J Polym Sci Polym Phys Ed v 16 n 5 May 1978 p 803-812
CODEN: JPLPAY

It has previously been shown that, under high stress and consequently at short times of failure, a major factor governing the environmental stress cracking (ESC) of polyethylene is the ability of a liquid environment to penetrate a growing fissure at a sufficiently high speed to maintain contact with the fracture front. In this earlier study, viscosity was shown to play a significant role in this kinetic effect. This paper presents experimental data which demonstrate that another property of the solid-liquid system influencing ESC under these high stress conditions is the spreading coefficient of the liquid on the polymer. The parameter defining the tendency of the liquid to wet the polyethylene. It is shown that the spreading coefficient can be considered as a force and this force in conjunction with atmospheric pressure constitutes the force necessary to drive the liquid into the growing crack. Method for calculating the spreading coefficient is included in Appendix 1. 5 refs.

DESCRIPTORS: (*POLYETHYLENES, *Wetting), (LIQUIDS, Surface Tension), (POLYMERS, Permeability), (CORROSION, Stress Corrosion Cracking), MATHEMATICAL TECHNIQUES.
IDENTIFIERS: SPREADING COEFFICIENT
CARD ALERT: 539, 801, 815, 921, 931

836375 ID NO. - E1780536375 ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE DRIP-IRRIGATION TUBING.

Chomoff, A. J.; Salovey, R.
Univ of South Calif, Los Angeles
Am Chem Soc Div Org Coat Plast Chem Prepr v 37 n 1 1977, 173rd Meet, New Orleans, La, Mar 20-25 1977. Publ by Am Chem Soc, Div of Org Coat and Plast Chem, Washington, DC p 769-773
CODEN: ACOCAO

Drip-irrigation is a recently developed sub-irrigation system whereby water, containing additives, is supplied directly to the soil around the roots of crops. This irrigation method is ideally suited for plastic applications in tubing and fittings. Water is premixed with fertilizers, insecticides and other additives and is pumped several times a day through thin-walled inflatable plastic tubing. Plastic materials adapted thus far for this application are polyethylene compounds ordinarily used in underground wire and cable coatings. It was observed that PE compounds optimized to resist cracking at a constant stress test are different from compounds optimized to resist cracking at constant strain (left double quote\$ bent strip right double quote\$ test). Tensile tests and burst tests were performed on long sections of tubing. Results are tabulated, plotted and discussed. 5 refs.

DESCRIPTORS: (*PIPE, PLASTIC, *Corrosion), (CORROSION, Stress Corrosion Cracking), (POLYETHYLENES, Agricultural Applications), (IRRIGATION, Piping Systems), MATERIALS TESTING,
CARD ALERT: 421, 539, 619, 817, 921

812733 ID NO. - E1780212723
**RADIATION CROSSLINKING OF POLYETHYLENE IN THE PRESENCE OF
 POLYMERIZABLE ADDITIVES.**

Zyball, A.
 Dtsch Kunstst Inst, Ger
 Kunstst Ger Plast v 67 n 8 Aug 1977 p 16-18 CODEN: KSGPA7
 Polyethylene can be crosslinked with the aid of high energy
 radiation, thereby improving certain properties such as heat
 distortion temperature, resistance to stress cracking and
 chemical resistance. The paper demonstrates how far the
 irradiation dosages can be reduced by incorporating the
 polymerizable additives in polyethylene, without affecting the
 degree of crosslinkage. Crosslinkage is demonstrated by
 plotting stress-strain curves at different temperatures, by
 long-term tensile tests and by measuring the heat distortion
 temperature. For figures and tables see German text. 33
 refs.

DESCRIPTORS: (*POLYETHYLENES, *crosslinking), RADIATION
 EFFECTS, (ELECTRONS, Applications), (POLYMERS, Additives), (MATERIALS TESTING, Tensile Tests), MONOMERS,
 IDENTIFIERS: POLYMERIZABLE ADDITIVES
 CARD ALERT: 421, 622, 701, 802, 804, 815

792408 ID NO. - E1771292409
ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE.
 Singleton, C. J.; Roche, E.; Gail, P. H.
 Case West Reserve Univ, Cleveland, Ohio
 J Appl Polym Sci v 21 n 9 Sep 1977 p 2319-2340 CODEN:
 JAPNAB

Deformation of polyethylene in environmental stress cracking
 (ESC) agents results in changes in both the mechanism of
 deformation and structure of the resulting drawn material.
 Stress-cracked failure surfaces are highly fibrillar, the
 fibrils having less elastic recovery than those in samples
 drawn in air. In thin films drawn in ESC agents, small blocks
 of the lamellae remain undrawn and attached to the fibrils
 drawn across micro-necks. The ESC agents are suggested to
 weaken the cohesion between the fibrils in samples drawn
 beyond yield as well as the cohesion between mosaic blocks or
 similar structural elements in the original lamellae as they
 are being reoriented to form the fibrils. The stress is thus
 supported by a number of independent, nonuniform fibrils
 rather than a coherent structure; the weakest of these
 fibrils fail in turn as the crack propagates through the
 sample. 8 refs.

DESCRIPTORS: (*POLYETHYLENES, *Weathering), (CORROSION,
 Stress Corrosion Cracking), (PLASTICS, Microstructure), (PLASTICS FILMS, Fracture)
 CARD ALERT: 423, 802, 815, 817

792408 ID NO. - E1771292408
ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE: A DYNAMIC

PHENOMENON AT HIGH STRESSES.

Schultz, J.; Shanahan, M. E. R.
 Univ du Haut-Rhin, Cent de Rech sur la Phys-Chim des
 Surfaces

Am Chem Soc Div Polym Chem Prepr v 18 n 2 Aug 1977: Spec
 Top in Polym Chem, Presented at Am Chem Soc Natl Meet, 174th,
 Aug 28-Sep 2 1977 p 355-359 CODEN: ACPPAY

In the reported experiments, the environmental stress
 cracking (E. S. C.) of a polyethylene has been studied as a
 function of stress, strain, temperature and the nature of the
 stress cracking agent using a simple test in which a
 polyethylene sample immersed in a liquid creeps in tension due
 to a constant applied force. Two distinct types of behavior
 can be observed. For long times to failure corresponding to
 low initial stresses, the liquid is in equilibrium with the
 polymer and a phenomenon due entirely to E. S. C. takes
 place. This behavior has been attributed to a reduction in
 the energy of cohesion of the solid due to the liquid
 environment. The influence of the viscosity of the stress
 cracking liquid is discussed in this paper in a quantitative
 manner. It is demonstrated that under conditions of high
 stress, an important parameter governing the overall time to
 failure of the polyethylene is the viscosity of the
 stress-cracking liquid. 6 refs.

DESCRIPTORS: (*POLYETHYLENES, *Weathering), (CORROSION,
 Stress Corrosion Cracking), VISCOSITY,
 IDENTIFIERS: ENVIRONMENTAL STRESS CRACKING
 CARD ALERT: 421, 631, 802, 817

768177 ID NO. - E1770968177
DETERGENT STRESS-CRACKING OF POLYETHYLENE.

Haward, R. N.; Owen, D. R. J.
 Univ of Birmingham, Engl
 Proc R Soc London Ser A v 352 n 1671 Feb 4 1977 p 505-521
 CODEN: PRLAAZ

The effects of detergents on polyethylene cracking are
 investigated. An electron micrographic study was carried out
 on polyethylenes cracked in the presence of an aqueous
 detergent (detergent stress cracking). It is shown that
 numerous small cavities and fibrils are formed on a scale
 small enough for the effect of surface tension to be
 significant. The role of the detergent in reducing surface
 tension and so promoting the fracture of polyethylene may
 therefore be understood. 46 refs.

DESCRIPTORS: (*POLYETHYLENES, *Decomposition),
 CARD ALERT: 815, 817

diameter. Certain aspects of the technology required for installation are also discussed with emphasis placed on the problems associated with fusion jointing the pipes. 7 refs.
DESCRIPTORS: *GAS PIPELINES, PIPE, PLASTIC, POLYETHYLENES, FRACTURE MECHANICS.
IDENTIFIERS: FUSION JOINTING
CARD ALERT: 522, 619, 817, 931

751484 ID NO. - E1770751484
ROLE OF INTERCRYSTALLINE LINKS IN THE ENVIRONMENTAL STRESS CRACKING OF HIGH DENSITY POLYETHYLENE.

Frayser, Paul D.; Tong, Paul Po-Luk; Dreher, William W.
Univ of South Miss, Hattiesburg
Polym Eng Sci v 17 n 1 Jan 1977 p 27-31 CODEN: PYESAZ
Results of dynamic mechanical spectroscopy, infrared spectroscopy, and tensile stress-strain data show that the nonionic surfactant Igepal CO-630, often used as a stress cracking agent, and water are absorbed by high density polyethylene to cause an internal stress relaxation of the intercrystalline tie molecules. The resulting molecular rearrangements produce changes in both the crystalline and amorphous regions. Thus, a molecular mechanism is proposed for the long-term aging process based on the results of accelerated aging in the presence of an environmental stress cracking agent. 10 refs.
DESCRIPTORS: (*POLYETHYLENES, *Stresses).
CARD ALERT: 815

751301 ID NO. - E1770751301
ESCR TESTING OF PE PIPE GOOD, BUT NOT FINAL ANSWER.

Funk, Dennis L.; Timmer, Roy C.
DuPont, Wilmington, Del
Pipeline Gas J v 204 n 3 Mar 1977 p 44-46, 48-49. 51
CODEN: PLGJAT
During the past few years, there has been intense interest in the environmental stress crack resistance (ESCR) of polyethylene pipe. The concern among users is that the presence of environmental stress cracking agents in fluids transported through polyethylene pipe, or in contact with the outside of the pipe, may impair its service life. This report delves into factors that influence stress cracking resistance and discusses whether some proposed standards might be practical. The applicability of a new test as a measure of quality control is also discussed. 4 refs.
DESCRIPTORS: (*PIPE, PLASTIC, *Testing). (POLYETHYLENES, Testing).
CARD ALERT: 619, 817

748847 ID NO. - E1770748847
USE OF LARGE DIAMETER POLYTHENE PIPELINES BY BRITISH GAS.

Greig, J. M.
Br Gas Eng Res Stn
Pipes Pipelines Int v 22 n 1 Feb 1977 p 16-24 CODEN: PPIIAU
The paper discusses the development of a PE system with large diameter pipes and the reasons for the change from established design concepts of ductile strength and stress cracking, to a design based on fracture propagation. Each of the three design concepts are examined leading to a determination of the design stress applicable to each pipe

712073 ID NO. - E1770212073
CRITERIA FOR THE CLASSIFICATION OF STRESS CRACKING PHENOMENA.

Pohrt, J.
Kunstst Ger Plast v 66 n 8 Aug 1976 p 11-14 CODEN: KSGPAT
If stress cracking is intentionally produced by means of aggressive media, one finds that there is a connection between the stress cracking effect and the condition of the test specimen as is also the case with impact strength (in air) using hole and double-V notches. This common dependence also indicates a connection between stress cracking resistance and impact strength. To experimentally clarify these relationships, tests were carried out on PVC, PP and PE in which the changes of the boundaries of crack formation in the medium and the impact strength in air on the one hand, are due to a change in the product and, on the other hand are due to modification of the state of the test specimen caused by processing or conditioning. For figures see German text. 9 refs.

DESCRIPTORS: (*PLASTICS, *Crack Propagation), (CORROSION, Stress Corrosion Cracking), (MATERIALS TESTING, Impact), POLYVINYL CHLORIDE, POLYPROPYLENE, POLYETHYLENES.
IDENTIFIERS: STRESS CRACKING OF PLASTICS
CARD-ALERT: 421, 423, 802, 817

68358 ID NO. - E176128358

KINETIC EFFECT IN THE ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE DUE TO LIQUID VISCOSITY.

Shanahan, M. E. R.; Schultz, J.
CMRS, Muihouse, Fr

J Polym Sci, Part A-2: Polym Phys v 14 n 9 Sep 1976 p 1567-1573 CODEN: JPLPAY

Results of an experimental study are presented. An interesting kinetic effect in the environmental stress cracking (E. S. C.) of PE has been observed, in which the liquid viscosity plays an important role. E. S. C. of a low density, high melt index PE due to silicone oils has been studied using constant load creep experiments. For relatively low stresses, it has been found that the time to fracture is independent of the viscosity of the silicone oil, all other factors being approximately equal. However, at high stresses, the time to fracture increases with increasing viscosity for a given stress. This effect has been shown to be due to the relative ease with which the liquid penetrates a growing crack and thus always be at the crack front. Times to fracture for viscous liquids at high stresses are longer since crack propagation continues partially with and partially without liquid contact, fracture rate being much slower when not in the presence of the liquid. 6 refs.

DESCRIPTORS: (*POLYETHYLENES, *Fracture), (CORROSION, Stress Corrosion Cracking), (LIQUIDS, Viscosity), (CHEMICAL REACTIONS, Reaction Kinetics), (POLYMERS, Crack Propagation), IDENTIFIERS: SILICONE OILS, CRACK FORMATION
CARD ALERT: 421, 631, 802, 815, 931

647543 ID NO. - E1760747543

ENVIRONMENTAL STRESS CRACKING IN HIGH PRESSURE POLYETHYLENE.

Filzek, P.; Sueselbeck, R.; Wicke, W.
Kunstst Ger Plast v 66 n 1 Jan 1976 Suppl p 11-13 CODEN: KSGPAT

The environmental stress cracking behavior of low density polyethylene (LDPE) in aqueous wetting agent solutions depends on, among other things, the type and concentration of the wetting agent. Other influencing factors are the mol. wt and the morphology of the plastic (degree of crystallization, orientation). The susceptibility of the material toward stress cracking increases with rising temperature and, at small deformations, with increasing loading of the specimens. Additives such as lubricants or pigments can increase the risk of stress cracking. Independent of the type of wetting agent. 9 refs.

DESCRIPTORS: (*POLYETHYLENES, *Corrosion), (CORROSION, Stress Corrosion Cracking), (CRYSTALS, Orientation),
CARD ALERT: 801, 802, 817

583332 ID NO. - E175128332

ESTIMATION FROM CENSORED, INTERVAL DATA OF THE MEDIAN

BREAKING POINT OF POLYETHYLENE SUBJECTED TO STRESS-CRACKING TESTS: A MONTE CARLO STUDY.

Denby, L.; Fowlkes, E. B.; Roe, R. J.
Bell Lab, Murray Hill, NJJ Appl Mech Trans ASME v 42 Ser E n 3 Sep 1975 p 607-612
CODEN: JAMCAV

Paper No. 75-WA/APM-9.
DESCRIPTORS: (*POLYETHYLENES, *Stresses), (MATHEMATICAL STATISTICS, Monte Carlo Methods),
CARD ALERT: 817, 921

561247 ID NO. - E1750961247

ESTIMATION FROM CENSORED, INTERVAL DATA OF THE MEDIAN BREAKING POINT OF POLYETHYLENE SUBJECTED TO STRESS-CRACKING TESTS: A MONTE CARLO STUDY.

Denby, L.; Fowlkes, E. B.; Roe, R. J.
Bell Lab, Murray Hill, NJASME Pap n 75-WA/APM-9 for Meet Nov 30-Dec 4 1975, 6 p
CODEN: ASMSAA

A Monte Carlo study has been carried out in order to study the properties of various alternative estimators for median breaking point of polyethylene specimens subjected to an environmental stress cracking test. Tables of bias, variance, and mean square error have been derived for different sample sizes, internal sizes, and levels of censoring data with the lognormal distribution as a model. These tables will aid in the design of experiments for efficient estimation of median breaking point. 8 refs.

DESCRIPTORS: (*POLYETHYLENES, *Stresses), (MATHEMATICAL STATISTICS, Monte Carlo Methods), (MATERIALS, Failure),
CARD ALERT: 817, 922

464784 ID NO. - E1741064784

ETHYLENE COPOLYMERS: TOUGH, FLEXIBLE THERMOPLASTICS.

Vaccari, John A.

Mater Eng v 80 n 2 Aug 1974 p 40-43 CODEN: MAEMBO

Physical properties, costs, and engineering applications are discussed for four classes of ethylene copolymers. Physical properties are tabulated for injection and blow-molding grades of ethylene-vinyl acetate (EVA), injection-molding, extrusion, and blow-molding grades of ethylene-ethyl acrylate (EEA), rotational-molding and blow-molding grades of ethylene-hexene (EH), and injection-molding grades of ethylene-butene (EB). Ethylene copolymers have many of the elastomeric qualities of rubber without the need for vulcanization, plus high flexibility without plasticizers. They are tough, resistant to stress cracking and retain their flexibility at very low temperatures. In discussing each copolymer class, such properties as temperature softening, chemical and solvent resistance, permeability to gases and liquids, and environmental stress-cracking are also considered.

DESCRIPTORS: *POLYMERS, (POLYETHYLENES, Physical Properties) (VINYL RESINS, Physical Properties), (ACRYLICS, Physical Properties), (POLYBUTENE, Physical Properties), (

POLYMERIZATION, Copolymerization).

IDENTIFIERS: ETHYLENE COPOLYMERS, THERMOPLASTICS

CARD ALERT: 817, 815

464756 ID NO. - E1741064756

MECHANISM OF ENVIRONMENTAL STRESS CRACKING IN POLYETHYLENE.

Singleton, C.; Roche, E. J.; Gell, P. H.

Case West Reserve Univ, Cleveland, Ohio

SPE, Annu Tech Conf, 32nd, Pap, v 20, San Francisco, Calif, May 13-16 1974 p 217-220. Available from SPE, Greenwich, Conn, 1974

Environmental stress cracking is a condition of failure exhibited by high polymers (esp. PE) which are subjected to polyaxial stresses while in contact with a non-solvent, surface active medium. If identical stresses are applied to the polymer in the absence of the stress crack inducing medium, failure does not occur. Results of extensive tests are presented and discussed. It has been found that stress cracking agents also affect the mechanism of deformation of PE samples drawn in tension. In order to observe the deformation process more closely, use has been made of single crystals as model systems. In contrast to the plastic deformation observed during uniform draw in air, numerous cracks are observed in the crystals drawn in both cracking agents. 7 refs.

DESCRIPTORS: (*POLYETHYLENES, *Corrosion), (CORROSION, Stress Corrosion Cracking), (PLASTICS, Crack Propagation), SURFACE ACTIVE AGENTS.

IDENTIFIERS: STRESS CRACKING MECHANISMS

CARD ALERT: 802, 804, 815, 817

464748 ID NO. - E1741064748

MORPHOLOGY STUDIES ON SOLVENT INDUCED CRYSTALLIZATION OF POLYETHYLENE TEREPHTHALATE.

Desai, Ajit B.; Wilkes, Garth L.

Princeton Univ, NJ

J Polym Sci, Part B: Polym Lett v 12 n 3 Mar 1974 p 113-119 CODEN: JPYBAN

In the reported investigation, the solvent-induced crystallization (SINC) of polyethylene terephthalate (PET) was studied by utilization of the methods of small-angle light scattering (SALS) and scanning electron microscopy (SEM). Experimental data are provided which show that, in the solvents of dioxane and acetone, an anisotropic spherulite morphology is induced in initially amorphous PET film. Other characteristics of the SINC process have also been revealed: local spherulite deformation, stress cracking, and cavitation caused by the diffusion front of the solvent. It is concluded that the results given represent the effects induced in the case where solvent diffuses into the polymer above the glass transition temperature of the polymer solvent system. 16 refs.

DESCRIPTORS: (*POLYESTERS, *Crystallization), (CRYSTALS, Structure).

IDENTIFIERS: POLYETHYLENE TEREPHTHALATE, MORPHOLOGY

CARD ALERT: 801, 802, 815

414821 ID NO. - E1740314821

WHY PLASTICS FAIL.

Lazar, Lawrence S.; Carroll, John

US Test Co, Hoboken, NJ

SPE, Eng Prop and Struct Div, Div Tech Conf, Tech Pap, Downingtown, Pa, Oct 9-10 1973 p 54-59. Available from SPE, Greenwich, Conn, 1973

This paper briefly discusses a number of case histories of product failures involving plastics to focus on the workings of a failure analyst. These are actual case histories of product failure which exacted a penalty from the parties involved. The case histories cover the gamut of items most plastic engineers know how to avoid, but didn't. They include \$EM DASH\$ 1. molding stresses (football helmets); 2. wrong plastics choice (chlorosulfonated PE hose); 3. environmental stress cracking (PVC siding); 4. poor mold design (plated ABS); and 5. weathering stresses (blow molding).

DESCRIPTORS: (*PLASTICS, *Fracture).

IDENTIFIERS: PLASTICS FAILURE

CARD ALERT: 421, 817

336294 ID NO. - E1730736294

ENVIRONMENTAL STRESS CRACKING OF POLYETHYLENE.

Schonhorn, Harold; Frisch, H. L.; Albarino, R. V.
Bell Lab. Murray Hill, NJ

J Polym Sci. Part A-2: Polym Phys v 11 n 5 May 1973 p 1013-1021 CODEN: JPDMAJ

Modification of the Griffith theory for the presence of liquids has been shown to explain some facets of the environmental stress cracking of polyethylene. In the absence of intercrystalline links and tie molecules, the authors find that one important factor is the interfacial tension generated between the spherulite boundary and the liquid environment. Judicious incorporation of silanes into polyethylene appear to reduce the tendency to stress cracking by modifying the interfacial tensions between the environment and the polymer. 10 refs.

DESCRIPTORS: (*POLYETHYLENES, *Crack Propagation), (CORROSION, Stress Corrosion Cracking).

CARD ALERT: 802, 815

325523 ID NO. - E1730525523

NOVEL CROSSLINKING METHOD FOR POLYETHYLENE.

Scott, H. G.; Humphries, J. F.
Dow Corning Corp, Midland, Mich

Mod Plast v 50 n 3 Mar 1973 4 p between p 82 and 87 CODEN: MOPLAY

A new means of crosslinking PE, now undergoing field evaluation in both Europe and the United States, allows high production rates without heavy capital investment in special equipment. It involves the addition of a silane to standard PE to form a crosslinkable polymer, and subsequent addition of catalyst in a masterbatch form. The PE is fabricated in the usual way, for example, by extrusion, and crosslinking takes place through a reaction involving water vapor. Like PE crosslinked by radiation or by organic peroxides, the resulting product possesses improved heat resistance, is nonmelting, and is completely resistant to environmental stress cracking. 12 refs.

DESCRIPTORS: (*POLYETHYLENES, *Crosslinking), (SILICON COMPOUNDS, (POLYMERS, Additives), (PLASTICS, Physical Properties).

CARD ALERT: 804, 815, 817, 931

303762 ID NO. - E1730103762

HIGH MOLECULAR WEIGHT POLYETHYLENE.

Anon

Europlast Mon v 45 n 8 Aug 1972 p 64-65

High molecular weight polyethylene is a term which encompasses a broad spectrum of polyethylenes in the 300,000 \$EM DASH\$ 1,000,000 plus molecular weight range. Properties which are determined by the molecular weight, increasing as the molecular weight is increased, are the ultimate tensile

strength; elongation at break; resistance to stress cracking; and notched Izod impact strength, particularly at low temperatures. Crystallinity is the most important factor in processing and applications of PE under consideration. The main processing technique for high molecular weight polyethylenes which can be moulded by conventional thermoplastics processing techniques is extrusion blow moulding. Major applicational outlet is for large containers which demand the increased toughness, stress cracking resistance and improved fatigue properties imparted by the higher molecular weight.

DESCRIPTORS: (*POLYETHYLENES, *Molecular Weight), (POLYETHYLENES, Physical Properties), (PLASTICS, Processing).

CARD ALERT: 815, 816, 817, 931

299184 ID NO. - E1721321188

CROSSLINKABLE ROTATIONAL MOLDING HIGH DENSITY POLYETHYLENE.

Carrow, G. E.

Phillips Petroleum Co, Bartlesville, Okla

SPE. Pap Annu Tech Conf 30th, Shaping the Future with Plastics, May 15-18 1972, Part 2, p 762-765

Properties and rotational molding of crosslinkable PE are discussed. Properly molded parts have outstanding environmental stress cracking resistance, impact strength and overall toughness. To optimize the part properties, molding techniques used with non-crosslinkable HDPE have to be slightly modified. The major areas which should be given consideration are oven temperature, cycle time, mold release and quality control testing. With a little care in mold design and mold construction rotational molded crosslinked parts with outstanding physical properties can be molded. To better evaluate part quality, special environmental and impact tests have been used. Crosslinkable HDPE resin has given the rotational molder a totally new material with which to work. Its impact, environmental stress cracking resistance and toughness open potential markets previously unavailable to the rotational molder.

DESCRIPTORS: (*POLYETHYLENES, *Molding), (POLYETHYLENES, Crosslinking), (PLASTICS, Mechanical Properties).

CARD ALERT: 421, 816, 817

296373 ID NO. - E1721318375
ENVIRONMENTAL STRESS CRACKING IN POLYOLEFINS.
 Marshall, G. P.; Linkins, N. H.; Culver, L. E.; Williams, J.

G. Imperial Coll., London, Engl
 SPE J v 28 n 9 Sep 1972 p 26-31 CODEN: SPEJAC
 Fracture mechanics parameter is a sensitive measure of the resistance of polyethylene to environmental stress cracking. The use of the stress intensity parameter and other allied parameters to explain and describe fracture processes is now standard practice in the metals field and is rapidly becoming accepted in the analysis of fracture in the more brittle glassy plastics. Results of an extensive test program are reported and discussed in practical terms. 12 refs.
 DESCRIPTORS: (*POLYOLEFINS. *Crack Propagation). (POLYETHYLENES. Fracture).
 CARD ALERT: 421, 423, 817

230471 ID NO. - E172X030471
Relations between processing method and stress cracking of pure and modified low density PE. (Sposoby przetworstwa a korozja naprzenicwa czystego oraz modyfikowanego polietylenu malej gestosci)
 KRZEMINSKI J; GNIADEK I
 Polimery v 16 n 5 May 1971 p 230-3 CODEN: POLIA
 The effect of injection and compression molding on the stress cracking resistance of pure low-density polyethylene and of this polyethylene modified by the addition of 1 to 5% polyisobutylene was measured and discussed. Experiments revealed that the highest stress cracking resistance has the injection molded specimens. The addition of polyisobutylene increases the stress cracking resistance of molded compression parts and decreases this resistance of injection molded items. 5 refs. In Polish.
 DESCRIPTORS: (*PLASTICS. *Molding). (PLASTICS. Polyethylene) (PLASTICS. Crack Propagation). (PLASTICS. Testing).
 CARD ALERT: 421, 817

227572 ID NO. - E172X027572
Polyethylene structures resistant to low temperature. (Een polyethyleenkonstruktie bestand tegen lage temperaturen)
 INKLAAR PA
 Belg Plast n 17 Mar 1971 p 43-7
 This is a report on design, production and applications of sandwiched structural plastics, based on PE, which are designed to be exposed to very heavy environmental conditions such as aggressive chemical medium and low temperatures. These items are made as structural foam, i. e., cellular plastics with integral skin. Results of chemical resistant tests are presented with particular attention to good stress-cracking resistance of materials under consideration. In Flemish.
 DESCRIPTORS: (*PLASTICS. *Polyethylene). (PLASTICS. Cellular

). (PLASTICS. Chemical Resistance). (CHEMICAL EQUIPMENT. Plastics).
 CARD ALERT: 802, 817

226197 ID NO. - E172X026197
Polybutylene. 'X' New generation 'X' plastic piping material
 HALE O
 Pipeline Gas J v 198 n 10 Aug 1971 p 52-3, 56
 Among outstanding features of polybutylene, a flexible polyolefin resin manufactured by Mobil Chemical Company for use in gas piping are the fact that it is a material with essentially no creep and no known stress cracking agents likely to be encountered in gas service. The material is collectable up to diameters of 4-in. and it has a strength regression line with a more gentle slope than that of polyethylene. Additionally, the strength regression line is displaced downward very gradually as the temperature is elevated, with the result that the strength regression line of polybutylene at 185 F is approximately the same as polyethylene at 73.4 F. It can be heat fused, and it has a high design stress rating.
 DESCRIPTORS: (*GAS PIPELINES. *Plastics Applications). (POLYMERS. Polybutene). (PIPE. PLASTIC. Testing).
 CARD ALERT: 522, 815, 817

215860 ID NO. - E172X015860
Life evaluation of insulated wires for submersible motors
 MIYASHITA T; OMADA T; KAMATA O
 Hitachi Rev v 20 n 5 1971 p 174-80 CODEN: HITAA
 A-c voltages at 50 Hz and 9 kHz were applied to polyethylene (PE), polyvinyl chloride (PVC) and fluorine resin coated wires submerged in water to study a water-treeing deterioration phenomenon known as 'water-treeing'. For PE wires, addition of a voltage stabilizer to the polyethylene did not result in improved characteristics; nor did treatment for resistance to stress cracking. Marked improvement was noted in PE wires provided with a metal shielding layer and those with an antitreeing layer. PVC was found resistant to treeing and the breakdown voltage of PVC-insulated wires did not fall much in the test. 10 refs.

DESCRIPTORS: (*ELECTRIC LINES. *Insulation). (ELECTRIC MOTORS IDENTIFIERS: SUBMERSIBLE MOTORS. ELECTRIC WIRE INSULATION TESTING)
 CARD ALERT: 705, 706

203938 ID NO. - E172X003938

Stress crack resistance of rotationally molded polyethylene

HOWARD HR

Union Carbide Corp. Bound Brook, NJ

SPE, 29th Annu Tech Conf, Washington, DC, Tech Pap v 17 May 10-13 1971 p 517-22

Report on the experimental study of factors affecting stress cracking of rotational molded PE parts. The stress crack test and the environmental stress rupture test methods were employed to determine the effect of resin variables on the two types of stress failures. All data were obtained on rotomolded specimens except for those injection molded specimens used for the comparison of the two processes. Results from experiments presented in tables and curves are discussed in terms of recommendations for rotomolders. The rotomolder is urged to investigate thoroughly all the requirements of a given application. 9 refs.

DESCRIPTORS: (*PLASTICS, *Molding). (PLASTICS, Crack Propagation). (PLASTICS, Polyethylene). (PLASTICS, Testing). (PLASTICS, Mechanical Properties).

CARD ALERT: 421, 816, 817

17977 ID NO. - E171X179777

How to avoid stress cracking in rotationally molded polyethylene parts

HOWARD HR

Union Carbide Corp. Bound Brook, NJ

SPE J v 27 n 7 July 1971 p 31-6 CODEN: SPEJJA

The effects of resin variables on rotationally molded polyethylenes in stress-cracking environments was studied. It was found that special modifications in roto-molding resins are essential to meet the requirements imposed by severe stress-cracking environments if the desired molding characteristics are to be obtained. Experimental data are tabulated, plotted in curves and discussed in terms of practical recommendations. It has been shown that careless processing and poor design can often cancel out good resin properties. 9 refs.

DESCRIPTORS: (*PLASTICS, *Crack Propagation).

CARD ALERT: 421, 816, 817

179610 ID NO. - E171X179610

Determination of stress-crack resistance of thermoplastics by using ball-indent method according to the DIN5349 Standard. (Beurteilung der Spannungsrisssbeständigkeit von Thermoplasten mit dem Kugelfeindruckverfahren nach DIN5349)

POHRT J

Gummi, Asbest, Kunstst v 29 n 9 Sept 1970 p 962, 964, 966, 968, 970, 972 CODEN: GAKSA

The theoretical and practical assessment of critical stressing of thermoplastics and its relation to stress-cracking ability of plastics are discussed and some practical

conclusions suggested. Principle of method for measurement of critical stress (crack formation mark) as function of the ball penetration is given followed by a description of standard procedure. Examples of measuring results obtained in applications for various plastic materials are given. Utilization of the method described for evaluation of welded plastics joints, particularly in cable sheets from polyethylene is demonstrated. 17 refs. In German.

DESCRIPTORS: (*PLASTICS, *Testing). MATERIALS TESTING Standards. (PLASTICS, Polyethylene).

CARD ALERT: 421, 817

124868 ID NO. - E171X024868

Sintered products with foam structure. (Gesinterde produkte mit schuimstruktur)

plastica v 22 n 11 Nov 1969 p 552-4 CODEN: PLASA

A short description is given of the rotational method for the production of polyethylene (PE) containers, between 17 liter and 13,000 liter. The sinter process, rotational molding, is done without any pressure, or any molecular orientation, and relatively little trouble with stress-cracking. The low density PE used is rather tough, but at low temperatures it becomes brittle. Therefore Vassen-Schoemaker Research Labs. developed a method for the production of a so-called Fusion Sandwich construction, made of three layers-solid PE, foamed PE and solid PE made in one production cycle. These products are extremely tough at room temperature and low temperatures. Some applications are described. In Dutch.

DESCRIPTORS: (*PLASTICS, *Cellular). (CONTAINERS, Plastics). (PLASTICS, Polyethylene).

CARD ALERT: 694, 817

117749 ID NO. - E171X017749

Environmental stress cracking of curved polyethylene specimens

OKAMOTO H; WATANABE H; SAKAKI Y

Proc 12th Jap Congr on Mater Res, Kyoto, Japan, Sept 1968 p 220-2

DESCRIPTORS: (*PLASTICS, *Crack Propagation). (PLASTICS, Stress). (POLYMERS, Polyethylene). (MATERIALS TESTING, Fracture).

IDENTIFIERS: ENVIRONMENTAL STRESS CRACKING

CARD ALERT: 000

DESCRIPTORS: (*PLASTICS, *Molds), (PLASTIC MACHINERY, Molding),
 CARD ALERT: 157

110225 ID NO. - E171X010225
 Experience- bases report on the fundamentals of the processing of polyethylene, (Erfahrungsbericht ueber Grundlagen zur Verarbeitung von Polyaethylen)
 BREUER H
 Kunststofftechnik v 9 n 1 Jan 1970 p 23-4 CODEN: KUNSB
 Several physical properties of molding materials together with processing factors affecting course of extrusion and injection molding of polyethylenes are discussed, tabulated separately for high density and low density materials. The processing parameters described include- melt index, molecular weight, stress- cracking resistance, stress- corrosion resistance, impact strength, density, crystal structure, yield stress, shear modulus, melt temperature, and after- crystallization. In German.
 DESCRIPTORS: (*PLASTICS, *Polyethylene), (PLASTICS, Processing), (PLASTICS, Extrusion), (POLYMERS, Physical Chemistry).
 CARD ALERT: 815, 816

108447 ID NO. - E171X008447
 Prevention of stress cracking of thermoplastics by irradiation crosslinking. (Zur Verhinderung der Spannungsrisbildung von Thermoplasten durch Strahlenvernetzung)
 LANGBEIN W; FISCHER H
 Kunststoffe v 60 n 4 Apr 1970 p 256-61 CODEN: KUNSA
 Stress cracking in plastics moldings which are simultaneously exposed to aggressive agents and stresses is discussed. One way of reducing the tendency towards stress cracking consists of irradiation with high energy rays. An experimental program is described in which samples of high- and low- density polyethylenes, polystyrene, nylon, rigid and flexible PVC and isotactic polypropylene were irradiated using Van- de- Graeff generator, followed by property measurement. Results from experiments are tabulated, plotted in curves and discussed in practical terms. 28 refs. In German.
 DESCRIPTORS: (*PLASTICS, *Irradiation), (PLASTICS, Testing), (PLASTICS, Crack Propagation).
 CARD ALERT: 817

005842 ID NO. - E170X005842
 Case for PTFE coating of molds
 BRUCKSHAW PJ
 Mass Production v 45 n 6 June 1969 p 66-7
 Considerable success has been achieved in coating of molds handling polyethylene. Nylon 6, ABS and PVC; in case of polyethylene rotationally molded, risk of environmental stress cracking is completely obviated with PTFE coatings as opposed to other mold release agents; negative factors are coating operation costs which, given reasonable efficiency, are regarded as negligible by those concerned.

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